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(12) United States Patent

Lamb et al.

(54) NON-SETTLING HYDROLYZED WHEY PERMEATE CONCENTRATE AND RELATED METHODS AND NUTRITIONAL COMPOSITIONS

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U.S.C. 154(b) by 0 days.

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- (60) Provisional application No. 61/162,164, filed on Mar. 20, 2009, provisional application No. 61/162,178, filed on Mar. 20, 2009.
- (51) Int. Cl. A23C 21/00 (2006.01)(2006.01)A23C 17/00 A23C 9/12 (2006.01)A23C 1/00 (2006.01)A23C 9/00 (2006.01)A23C 9/154 (2006.01)A23C 9/16 (2006.01)A23C 21/02 (2006.01)A23C 1/12 (2006.01)

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(52) U.S. Cl.

(2013.01); *A23C 21/02* (2013.01); *A23C 21/026* (2013.01); *Y02P 60/875* (2015.11)

(58) Field of Classification Search

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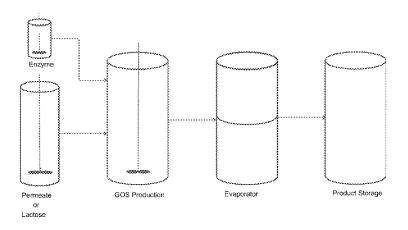
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(57) ABSTRACT

The present invention includes a method of producing non-settling hydrolyzed whey permeate with an enzyme, the non-settling hydrolyzed whey permeate concentrate, and nutritive additives and foods made therefrom.

8 Claims, 42 Drawing Sheets



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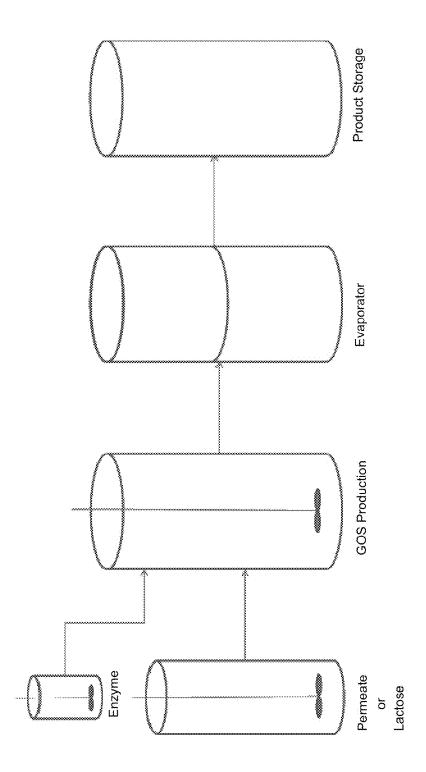


Figure 1

LAC-090209A-0.5% Novo, pH6.5

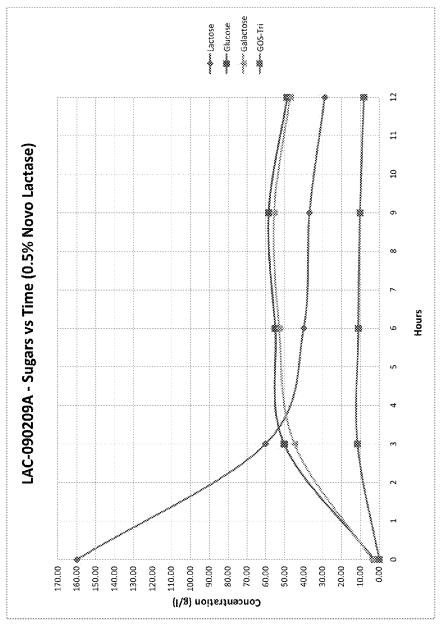


Figure 2

LAC-090209B-0.2% Novo, pH6.5

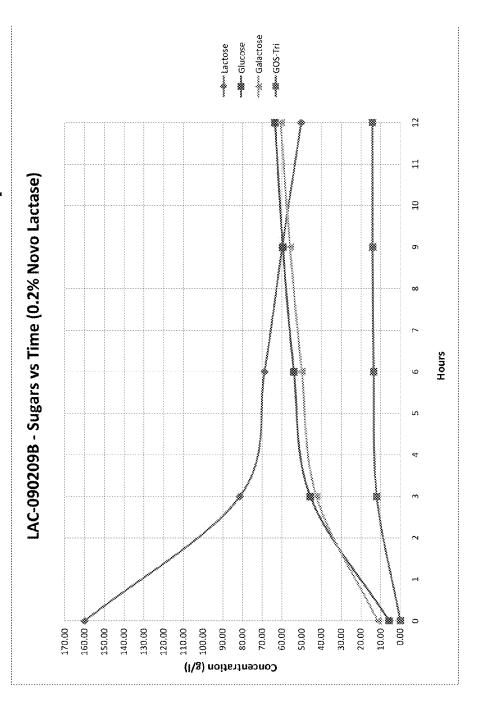


Figure 3



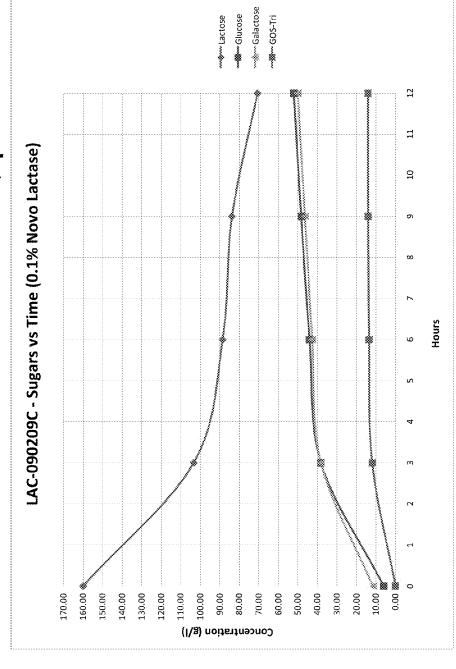


Figure 4

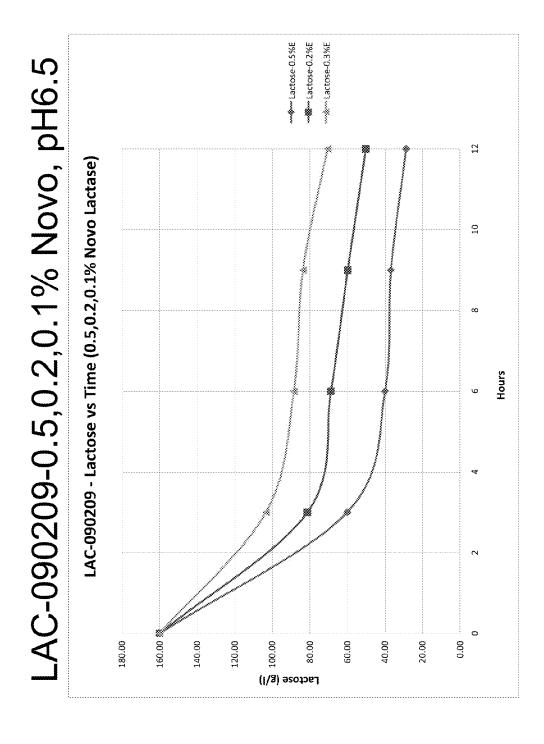


Figure 5

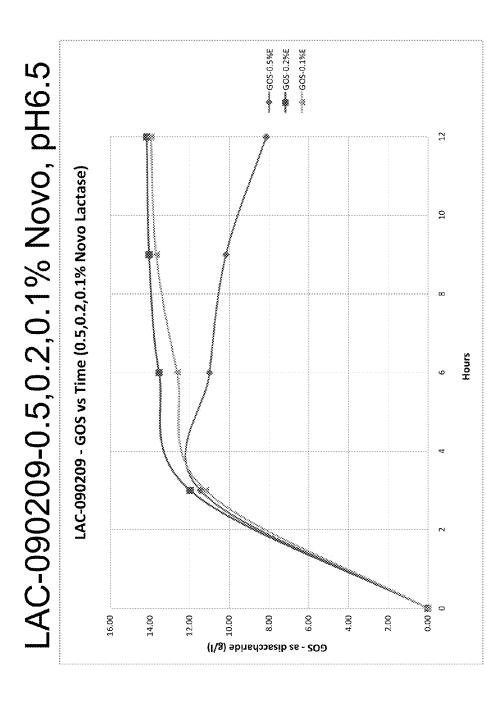


Figure 6

LAC-090209 A, B, C

pH 6.5 - 12 hr Hydrol - 80% Solids

A. 0.5 % Novo Lactase

B. 0.2 % Novo Lactase

C. 0.1 % Novo Lactase

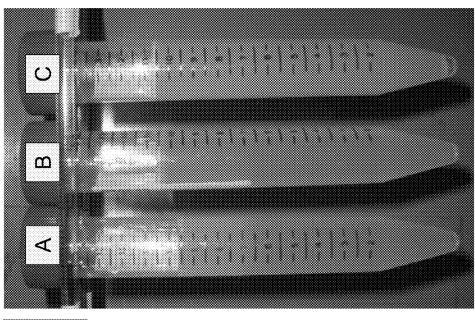


Figure 7

| LAC Plant I rials 1 & Z HPLC Data | als I & | ノロアし | , Data |
|--|----------------|----------------|----------------|
| | Sample | Plant Trial #1 | Plant Trial #2 |
| | Lactose Source | 20% Permeate | 20% Permeate |
| | Enzyme Level | 0.50% | 0.50% |
| | Hrs | Concentrate | Concentrate |
| Lactose (+GOS Disaccharide) (gm/l) | 1st rep | 82.02 | 75.18 |
| | 2nd rep | 82.30 | 75.21 |
| | AVG | 82.16 | 75.19 |
| Glucose (gm/I) | 1st rep | 40.83 | 44.11 |
| | 2nd rep | 40.96 | 44.14 |
| | AVG | 40.90 | 44.13 |
| Galactose (gm/l) | 1st rep | 41.56 | 46.22 |
| | 2nd rep | 41.65 | 46.24 |
| | AVG | 41.60 | 46.23 |
| GOS-Trisaccharide (gm/l) | 1st rep | 12.69 | 13.77 |
| | 2nd rep | 12.74 | 13.77 |
| | AVG | 12.72 | 13.77 |
| %Lactose Hydrolyzed (based on Glucose-160 start) | (molar) | 48.56% | 52.40% |
| | | | |

Figure 9

| w | | (17-20%Perm-pH6.5 0.5% lactase at 95 F for 12 hours - Evap) | | | |
|-------------|--|---|--------------|------|---------------|
| 1 | | To develop piant-scale process for 75% (solids) hydrolysis as molasses | | | |
| 5 75 | 09-003,#6 | 09-003, #6 freplacement | | | |
| | | pasteurize the permeale | | | |
| | | hydrolyze the 17-20% permeate with lackase at pH 6.5, 95 F for 12+ hours | | | |
| | | evaporate the hydrolysis to 70-80% as finished products through MVR and TVR | | | |
| | 35,500 | 35,500 (lbs Permeate with 17-20% solids (Lising 17-20% permeate off the RVD) | | | |
| | 32.5 | 32.5 ilbs Lactozyme | | | |
| | Pasteurized | Pasteurized 17-20% Permeate - 95 F, pH 6.5 with 0.5% Lacozyme3000L (1 dosing) for 12+ | | 1 | |
| | hrs, then ev | firs, then evaporation to 75% solids | & | Z | a |
| | | Pasieurize the permeate - heat up to 150 F/1 fir (160 F/20-30 min) and cold down | | | |
| | | to 95 Fin a A tank | | | |
| | | OP and sandize C12. | | | |
| | | Transter the pasteurized permeate into C12 | | | 10:30 AM |
| | | Adjust pH to 6.5 with 50% caustic | 97.73 | 6.51 | |
| | | Add 32.5 lbs of Lactozyme3000L to 32.5 lbs of water and mix well. Add to C12. | | ~~~~ | 10:50 AM |
| | | Check pH and adjust pH if necessary | | 6.35 | 6.36 2:50 PM |
| | | Check pH and adjust pH if necessary | | 633 | 430 PM |
| | | Check pH and adjust pH if necessary | | 628 | 6.26 (0.50 PM |
| | | Finish the hydrolysis at 95 F for 12 hrs if pH <5.9. If pH >5.9 hydrolyze for another 4 | | | |
| | | Nous | | | evaporated |
| | | Evaporate to 70-80% Solids through MVR and TVR | | | 75% |
| | | Fill the 70-80% hydrolysis in totes at 140 F | | | |
| | Annual Contraction of the Contra | | | | |

| To determine the effect of hydrolysis of 20% Perm with Noro Lactosymposition to 10.5 to 2 and 0.1% or Permease Solids, et 150.0 per vision Leaders | euce: | | | | | |
|--|----------|------------------------------------|--|----------------|------------------|----------|
| 1700 gam Veter 1.20 gam Dried Permeate 2.21 gam Dried Permeate 2.21 gam Dried Permeate 2.21 gam Dried Permeate 2.22 gam Dried Permeate 2.22 gam Dried Permeate 2.23 gam Dried Permeate 2.24 gam Dried Permeate 2.24 gam Dried Permeate 2.24 gam Dried Permeate 2.24 gam Dried Permeate 2.25 gam Dried Permeate 2.24 gam Dried Permeate 2.24 gam Dried Permeate 2.24 gam Dried Permeate 2.24 gam Dried Permeate 2.25 gam Dried Permeate 2.24 gam Dried Permeate 2.25 gam Dried Permeate 2.27 gam Dried Permeate 2.27 gam Dried Permeate 2.27 gam Dried Permeate 2.28 gam Dried Permeate 2.29 gam Dried Permeate 2.20 gam Dried Permeate 2.20 gam Dried Permeate 2.21 gam Dried Permeate 2.21 gam Dried Permeate 2.22 gam Dried Permeate 2.23 gam Dried Permeate 2.24 gam Dried Permeate 2.25 gam Dried Permeate 2.25 gam Dried Permeate 2.27 gam Dried Permeate 2.27 gam Dried Permeate 2.27 gam Dried Permeate 2.27 gam Lectorymaggoo to 20% Solids 2.27 gam Lectorymaggoo to 20% Solids 2.27 gam Lectorymaggoo to 20% Solids 2.27 gam Lectorymaggoo to 20% Solids samples 2.27 gam Lectorymaggoo t | | | | | | |
| 1700 grm Veter 1700 grm | | To determine the effect | t of hydrolysis of 20% Perm with Novo Lactozyme3000 at 0.5, 0.2 and 0.1% on Permeate Solids, at | | | |
| 1700 pg my brear | U | SOC, PHO S for TZRIS, R | ollowed by adjusting pH to 4.0 with conc HCl, followed by evaporation to 60 & 60% solids. | _ | | |
| 4.25.0 gm More Lestrozyme 3000. 1.00.0 gm Water 1.00.0 gm Wat | ↲ | 1700.0 gm Water | | _ | | |
| 2.13g MV ob Lactozyme 3000L 1700.0 gm VM ster 425.0 gm Dried Permeate 625.0 gm Dried Permeate 626.0 g | | 425.0 gm Dried Permeate | | | | |
| 1700 gpm Vertice 1700 g | | 2.13 gm Novo Lactozyme 30 | 7000 | | | |
| 1700 og m/ PELESOO 1700 og | <u></u> | 1700.0 gm Water | | | | |
| 0.05 9m DFL5000 1700 9m Valenteerine | L. | 425.0 qm Dried Permeate | | | | |
| 1700 gm Weter 4.50 gm Died Permaste 4.50 gm Chee Permaste 4.50 gm Chee Permaste 1.52 to 50 gm Evap 16.0 5% Solids 1.60.3 to 50 gm Evap 16.0 38 gm to 133 gm 1.60.3 to 50 gm Evap 16.0 38 gm to 133 gm 1.60.3 to 50 gm Evap 16.0 38 gm to 133 gm 1.60.3 to 50 gm Evap 16.0 38 gm to 133 gm 1.60.3 to 50 gm Evap 16.0 18.0 gm 1.60.1 to 50 gm Evap 16.0 gm 1.60.1 to 50 gm Evap 16.0 gm 1.60.1 to 50 gm In 18.0 gm 1 | | 0.85 gm DFL5000 | | | | |
| 10.13 gim Died Permeate 0.43 gim Died Permeate 0.43 gim Died S0% Solids Evap 125 gim to 140 gim Evap 125 gim to 135 gim to 135 gim 174.63 to 50 gim Evap 125 gim to 135 gim 174.63 to 50 gim Evap 120.38 gim to 135 gim 174.63 to 50 gim Evap 10 70% Solids 174.63 to 50 gim Evap 10 70% Solids 174.63 to 50 gim Evap 10 70% Solids 174.63 to 50 gim Evap 10 10 gim to 50 | <u>l</u> | 1700.0 gm Water | | | | |
| Evap 105 gm DFL5000 Evap 105 gm to 140 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm to 150 gm Evap 105 gm to 150 gm t | <u> </u> | 425.0 am Dried Permeate | | | | |
| Evap to 50% Solids | Ļ | 0.43 gm DFL5000 | | | | |
| 126 to 50 gm Evap 125 gm to 140 gm Evap to 60 gm Evap 150.38 gm to 133 gm Evap to 60 gm Evap 150.38 gm to 133 gm 174.63 to 50 gm Evap 170.65 bilds 175.65 gm disk 120 bilds 120 b | | | | (20%)*(125)=(| £0%)*X | |
| Evap to 60% Solids Evap to 10% Solids | | 15 | | X=(20)*(125)/(| 50)= | S |
| 150.38 to 50 gm Evap 150.39 gm to 133 gm 174.63 to 50 gm Evap 170.49 Solids Evap to 70% Solids Evap 200 gm to 50 gm to 136 gm Evap 170.50 gm to 50 gm to 50 gm Evap 170.63 gm to 50 gm to 50 gm Evap 170.63 gm to 50 gm to 50 gm Add 1700 gm to 50 gm to 50 gm Add 4700 gm to 50 gm to 50 gm Add 41700 gm to 50 gm to 50 gm Mix at 170.63 min - Then 0.5%Lactozyme3000 at 35C,pH6.5(wHCl)-Then pH4.0(wHCL), Evap to 60 & 80% Solids Add 41700 gm to 50 gm to 50 gm to 50 gm to 50 gm to 60 gm | | | | RE 051/1/2001 | X*170US/=1 | 1000 |
| Evap to 70% Solids T4.63 to 50 gm Evap 174.63 gm to 136 gm Evap 200 Solids Evap 200 Solids Evap 200 Solids Evap 200 Solids Evap 200 gm to 65 gm 20% Permeate - 70C, 30 min - Then 0.5%Lactozyme3000 at 35C, pH6.5(w/HCl)-Then pH4.0(w/HCl), Evap to 60 & 80% Solids Add 1700 gm DI water to glass reactor in water bath such that reaction mix temp is 75C Add 1700 gm DI water to glass reactor while mixing until all Permeate is weited and evenly suspended Mix at 70C for30 min Cool to 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm 1.8 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab)- Add 2.13 gm 1.6 so 30% solids a fler Lactozyme3000 addition sample for tolovap to 60% solids it sediment rolovap second sample to 60% solids. Use designated amount above (200gm - 50gm for 60% solids in 60% solids in 60%) - Label 1.AC-090209-A3.40(60, A6.90(60, A6.9 | | 38 to 50 gm Evap 150.38 gm to 133 | | X=(20)*(150.3 | 8)/(60)= | 8 |
| Fiva D 174.63 gm to 136 gm Evap 174.63 gm to 136 gm Evap 174.63 gm to 150 gm Evap 108 Solids 2006 bermeate - 106.73 gm int - 174n 0.53/Lactozyme3000 at 33C, phi6 Siw/HCI)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids Add 1700 gm Di water to glass reactor in water tenth such that reaction mix temp is 75C. Add 425 gm dried Permeate slowly to reactor while mixing until all Permeate is wetted and eventy suspended Add 1700 gm Di water to glass reactor in water that the second plat and control and control and control and a 6.5 with NaOH or HCI while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Solids amples for 80% and 60% Evap drop pH to 4.0 with Conc HCl and Rotvoap flask on 60% solids. It sediment rotovap second sample to 60% solids. Jee designated amount above (200gm | | Evan to 70% Solids | | CA 1771-17001 | V=(7007)=(| |
| Evap to 80% Solids 20% Permeate - 70C, 30 min - Then 0.5% Lactozyme3000 at 35C, pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60.8.80% Solids 20% Permeate - 70C, 30 min - Then 0.5% Lactozyme3000 at 35C, pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60.8.80% Solids Add 4150 gm died fermeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Add 425 gm died fermeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Add 425 gm died fermeate slowly to reactor while mixing Add 425 gm died fermeate slowly to reactor while mixing Add 2.13 gm Leatozyme3000 to 50 min water in beaker and mix well Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Leatozyme3000 to 20% Permeate at 35C while mixing Add 3.6. 9 and 12 hrs after Lactozyme3000 addition samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCI and Roveya first to 80% solids In conc sample to 80% solids on 80 and 80% solids samples in cold and take pics at 1 day and periodically over next week Pre-weigh 1 liter Rotovap flask Tensfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture profits to 8% solids to sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Add 1700 gm Di water to glass reactor; white that reaction mixture to 80% solids solids on sample profit | | 33 to 50 gm Evap 174.63 gm to 136 | | X=(20)*(174.6 | 3)/(70)= | S |
| 100% Permeate - 70C, 30 min - Then 0.5% Lactozyme3000 at 35C, pH6 5(wrHCl). Then pH4.0(wrHCL), Evap to 60 & 80% Solids Add 1700 gm Di water to glass reactor in water bath such that reaction mix temp is 75C Add 4750 gm Di water to glass reactor while mixing until all Permeate is wetted and evenly suspended Mix at 70C for30 min Cool to 35C while mixing Add well mixed def ermeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 70C for30 min Cool to 35C while mixing Add well mixed Lactozyme3000 to 50 ml water in beaker and mix well Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing At 0. 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) LAC-090209-A0,A3,A6,A9A.12 At 0. 3, 6, 9 and 12 hrs after Lactozyme3000 addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rokovap first to 80% solids - I no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 60% solids - I no sediment, Stop and do not rotovap to 60% solids - I no sediment, Stop and and A12-80/80 for 60% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week Record weight of Rotovap flask Record weight of Rotovap flask Record weight of Rotovap flask Tansfer Calculated gm (see abova) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher 20% solids reaction while mixing until all Permeate is wetted and evenly suspended Add 4700 gm Di water to glass reactor in water bats buch that reaction mix lenp is 75C Add 4700 min - Then 0.2%Lactozyme3000 at 35C,pH6.5/WHCD)-Then PH4.0(wHCL), Evap to 60 & 80% Solids Add 4700 gm Di water to glass reactor in water bats buch that reaction mix lenp is 75C Add 4700 min - Then 10.2%Lactozyme3000 at 35C,pH6.5/WHCD)-Then PH4.0(wHCL), Evap pro 60 & 80% Solids Add 4700 gm Di water to glass reactor in water ba | _ | Piloo /600 cj xext | | 10003110000 | | 1 |
| Add 120 gm to 30 gm to 30 gm 120% Permeate - 10C, 30 min - Then 0.5%Lactozyme3000 at 35C,pH6.5[w/HCl]. Then pH4.0(w/HClL), Evap to 60 & 80% Solids Add 1700 gm to 30 min - Then 0.5%Lactozyme3000 at 35C,pH6.5[w/HCl]. Then pH4.0(w/HClL), Evap to 60 & 80% Solids Add 1700 gm to 30 water to glass reactor in water balt with receive mix terms and evenly suspended Mix at 700 6/030 min Cool to 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing mixiture DO NOT do Viscosity on 60 and 80% solids and 412.80/60 for 80% and 60% solids. Use designated amount above (200gm -> 50gm for 80% and 160 costly on 60 and 80% solids samples Slore 80% and fi necessary 60% solids samples Slore 80% and fi necessary 60% solids samples Slore 80% and fi necessary 60% solids samples Acold 2.25 gm died permeate from remaining "20% Solids" mixture DO NOT do Viscosity on 60 and 80% solids samples in cold and take pics at 1 day and periodically over next week Pre-weigh 1 liter Rotoxap flask Record weight of Rotoxap flask Do NOT 30 weight of Rotoxap flask Acod 2.25 gm died Permeate slowly to reactor while mixing until all Permeate is weited and evenly suspended Add 4.25 gm died Permeate slowly to reactor while mixing until all Permeate is weited and eve | _ | 1 | |)=(002)_(%07) | ¥.(%09 | |
| 20% Permeate - 70C, 30 min - Then 0.5% Lactozyme3000 at 35C, pH6.5 (wMCI)-Then pH4.0 (wMHCL), Evap to 60 & 80% Solids Add 425 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended Add 425 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 70C for30 min mixing Add 425 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 70C for30 min mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing Add 425 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended Add 426 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended Add 426 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended Add 427 gm dried Permeate slowy to reactor while mixing until all Permeate is wetted and evenly suspended | 3] | o 50gm Evap 200 gm to 50 gm | | X=(20)*(200)/(| 80) = | 25 |
| Add 1700 gm DI water to glass reactor in water beth such that reaction mix temp is 75C Add 425 gm dided Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 70C for30 min Cool to 35C while mixing Add 425 gm dided Permeate slowly to reactor while mixing Add 21 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add 8.3 (6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) LAC-090209-A0,A3,A6,A9,A12 At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition - Take a samples for 80% and 6100 pH to 4.0 with Conc HCl and Rotovap first to 80% solids. If no sediment show (200gm -> 50gm for 80% solids. If sediment rotovaps second sample to 60% solids. Use designated amount above (200gm -> 50gm for 80% solids, respectively. DO NOT do Viscosity on 60 and 80% solids samples Store 80% and if necessary 60% solids samples Store 80% and if necessary 60% solids samples Store 80% and if necessary 60% solids samples Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for sight stells. Do % Solids tests Add 475 gm and and an eventy that reaction mixture are such and eventy suspended Add 475 gm and and he measured in water bath such that reaction mix temp is 75C Add 475 gm and and Permeate slowly to reactor while mixing until all Permeate is wetted and eventy suspended | A 20 | Permeate - 70C, 30 min - Then 0.5% | "Lactozyme3000 at 35C, pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids | Ē | _ | Time |
| Add 425, gm dried Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 70C for30 min. Mix at 70C for30 min. Cool to 35C while mixing Add 22 gm dried Permeate stown to reactor while mixing Add 2.13 gm Lactozyme3000 to 50 ml water in beaker and mix well Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing At 0. 3. 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab)- LAC-090209-A0,A3,A6,A9,A12 At 0. 3. 6, 9 and 12 hrs after Lactozyme3000 addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 60% solids - L9e designated annount above (200m —- 50gm for 80% solids. If sediment rotovap second sample to 60% solids - L9e designated annount above (200m —- 50gm for 80% solids. It sediment rotovap second sample to 60% solids respectively. DO NOT do Viscosity on 60 and 80% solids samples in cold and take pics at 1 day and periodically over next week Pre-weight of lifer Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids tests Do % Solids on sample from remaining "20% Solids" mixture Do % Solids tests Do % Solids tests Add 475g min - Then 0.2%Lactozyme3000 at 35C, pH6.5(wHCI). Then pH4.0(w/HCL), Evap to 60 & 80% Solids Add 475g min - Then 0.2%Lactozyme3000 at 35C, pH6.5(wHCI). Then pH4.0(w/HCL), Evap for one and solwy to reactor while mixing until all Permeate is wetted and evenly suspended Add 475g min - Then 0.2%Lactozyme300 while mixing until all Permeate is wetted and evenly suspended | | Add 1700 gm DI water t | to glass reactor in water bath such that reaction mix temp is 75C | | Н | |
| Mix at 70C for 35C while mixing Cool to 35C while mixing Adduct 21 gam Lactozyme3000 to 20% Permeate at 35C while mixing Adduct 21 gam Lactozyme3000 to 20% Permeate at 35C while mixing Add Add 21 gam Lactozyme3000 to 20% Permeate at 35C while mixing Add Add 21 gam Lactozyme3000 to 20% Permeate at 35C while mixing Add 40 3.5, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab)- LAC-090209-A0.A3.A6.A9.A1.2 At 0. 3, 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 60% solids - Jes designated amount above (200gm -> 50gm for 80% solids. If sediment rotovap second sample to 60% solids - Ad-8060, Ad-8060 ond -A12-8060 for 80% and if necessary 60% solids samples LAC-090209-A3-3060, A6-8060, A9-8060 and -A12-8060 for 80% and if necessary 60% solids respectively. DO NOT do Viscosity on 80 and 80% solids samples in cold and take pics at 1 day and periodically over next week Pre-waden 1 littler Rotovap flask Transfer Calculated 9m (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture to % solids tests Do % Solids tests Do % Solids tests Do % Solids tests Do % Solids tests Add 475 gam Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 475 gam Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 475 gam dived Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended And 475 gam Di water to glass reactor in water beth such that reaction mixture is wetted and evenly suspended | _1 | Add 425 gm dried Perm | neate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | | |
| Cool to 35C while mixing Cool to 35C while mixing Record pH and control pH at 6.5 Record pH and control pH at 6.5 Add 2.13 gm Lactozyme3000 to 50 ml water in beaker and mix well Add 2.13 gm Lactozyme3000 to 50 ml water in beaker and mix well Add 2.13 gm Lactozyme3000 to 20% Permeate at 35C while mixing Ad 0. 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab)- LAC-090209-40,A3-A6,A9,A12 At 0. 3, 6, 9 and 12 hrs after Lactozyme3000 addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotovap first to 80% solids. It sediment rotovaps second sample to 60% solids. If sediment rotovaps second sample to 60% solids. If sediment rotovaps second sample to 60% solids. If sediment rotovaps second sample to 60% solids. In sediment rotovaps second sample to 60% solids. A3-80/60, -A5-80/60 and 40/2.80/60 for 60% and if necessary 60% solids, respectively. DO NOT do Viscosity on 60 and 80% solids samples Store 80% and if increasany 60% solids samples Store 80% and if increasany 60% solids and add and lake pics at 1 day and periodically over next week Pre-weigh 1 lifer Rotovap flask Record weight of Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids on sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Add 472 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 472 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 472 gm of the Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended And 472 gm of the Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | Mix at 70C for30 min | | | | |
| Add use the to 6.5 with Nach or HCI while mixing Record pH and control pH at 6.5 Record pH and control pH at 6.5 Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add 4.0 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab)- LAC-090209-A0,A3,A6,A9,A12 At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotovap first to 80% solids. If sediment rotovap second sample to 60% solids. Use designated amount above (200gm - 5 50gm for 80% solids. If sediment rotovap second sample to 60% solids. Ad-80060. Ad-80060. Ad-80060 and 40.2-80.60 for 80% and if necessary 60% solids, respectively. DO NOT do Viscosity on 60 and 80% solids samples in cold and take pics at 1 day and periodically over next week Record weight it filer Rotovap flask Record weight it fler Rotovap flask Record weight of Rotovap flask Record weight of Rotovap flask Record weight of Rotovap flask Do % Solids tests Add 475 gm of weight of Rotovap flask reactor in water bath such that reaction mix lemp is 75C Add 475 gm of weight of Rotovap flask reactor in water bath such that reaction mix lemp is 75C Add 475 gm of well Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Add 475 gm of Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | Cool to 35C while mixing | Ďi. | | | |
| Record pH and control pH at 6.5 Record pH and control pH at 6.5 Add 2.13 gm Lactozyma3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyma3000 to 20% Permeate at 35C while mixing At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - LAC-090209-A0,A3,A6,A9,A12 At 0, 3, 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl sample to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 60% solids - Lace designated amount above (2009m> 50gm for 80% solids. If sediment rotovap second sample to 60% solids - A6-80/60_A-A6-80/60_A-A10/60 for 80% and if necessary 60% solids respectively. DO NOT do Viscosity on 80 and 80% solids samples in cold and take pics at 1 day and periodically over next week Pre-weight it lifer Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids tests Do % Solids tests Do % Solids tests Do % Solids tests Add 4750 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 4750 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 4750 gm Di water to glass reactor while mixing until all Permeate is wetted and evenly suspended Add 4750 gm Di water to glass reactor while mixing until all Permeate is wetted and evenly suspended | | Adjust pH to 6.5 with Na | aOH or HCI while mixing | | | |
| Add 2.13 gm Lactozyme3000 to 50 ml water in beaker and mix well Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing At 0. 3. 6. 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - LAC-090209-A0,A3,A6,A9,A12 At 0. 3. 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. Use designated amount above (200gm -> 50gm for 80% and 150.38gm -> 50gm for 60%) - Label LAC-090209-A3-80160, -48-80160 and -412-80160 for 60% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week Pre-waight it filter Rotovap flask Broon Weight of Rotovap flask Record weight of Rotovap flask Transfer Calculated 9m (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids tests Do % Solids tests Do % Solids on sample from remaining "20% Solids" mixture Add 1700 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 4120 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Mix at 720. As an and and evenly suspended Mix at 720. As an and a such that reaction mix lemp is 15C Mix at 720. As an and a such that mixing until all Permeate is wetted and evenly suspended | | Record pH and control | I pH at 6.5 | | | |
| Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing Ad 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab)- At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotozyme3000 addition - Take a samples for 80% and 150.38pm -> 50gm for 60%) - Label LAC-199209-A3-80/80. A64.80/80. A64.80/80 and -412-80/80 for 80% and if necessary 60% solids. Insepectively. DO NOT do Viscosity on 60 and 80% solids samples Store 80% and if necessary 60% solids samples Bo % Solids on sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Add 4120 min - Then 0.2% Lactozyme3000 at 35C,pH6.5(wHCI)-Then pH4.0(wHCL), Evap to 60 & 80% Solids Add 4120 m Di water to glass reactor in water bath such that reaction mix lemp is 75C And 4120 m De media formed to glass reactor in water bath such that reaction mix lemp is 75C And 4120 m De media formed to mixture for while mixing until all Permeate is wetted and evenly suspended | | Add 2.13 gm Lactozyn | me3000 to 50 ml water in beaker and mix well | | | |
| At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) LAC-090209-A0,A3,A6,A9,A12 At 0, 3, 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCl and Rotovap first to 80% solids. If sediment rotovap second samples for 80% and 150.38gm - 50gm for 80% and 150.38gm - 50gm for 80% and 160.38gm - 50gm for 80% solids. It sediment rotovap second sample to 60% solids. Label LAC-090209-A3.80060. A8-20160 and -A12-80060 for 80% and if necessary 60% solids for 60%). Label LAC-090209-A3.80060. A8-20160 and -A12-80060 for 80% and if necessary 60% solids respectively. DO NOT do Viscosity on 60 and 80% solids samples in cold and take pics at 1 day and periodically over next week Pre-weigh 1 lifer Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids tests Do % Solids tests Do % Solids to sample from remaining "20% Solids" mixture Do % Solids to sample from remaining "20% Solids" mixture Do % Solids to sample from remaining "20% Solids" mixture Add 475 gm Di water to glass reactor in water beth such that reaction mix temp is 75C Add 475 gm Di water to glass reactor in water beth such that reaction mix temp is 75C MAN AT 200 And 1700 gm Di water beth such that reaction mix temp is 75C MAN AT 200 And 1700 gm Di water beth such that reaction mix temp is 175C | | Add well mixed Lactoz | rzyme3000 to 20% Permeate at 35C while mixing | | | |
| At 0. 3. 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCI and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 80% solids - A6-80160_A-A | | At 0, 3, 6, 9 and 12 hrs | after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - | | | |
| At 0. 3. 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCI and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 60% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second sample to 60% solids - 46.806.00 and -412.800.00 - > 50gm for 60% solids - 50gm for 60% and 10 cessary 60% solids amples in cold and take pics at 1 day and periodically over next week Pre-waight tiller Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids tests Do % Solids tests Do % Solids on sample from remaining "20% Solids" mixture Add 1700 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C Add 1700 gm Di water to glass reactor in water beth such that reaction mix lemp is 75C May a 720 for and weigh of the Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended May a 720 for an armonia water beth such that reaction mix lemp is 75C May a 720 for an armonia water beth such that reaction mix lemp is 75C | | LAC-090209-A0,A3,A6, | 3,A9,A12 | | | |
| Art 0, 5, 9, 2 and 12 first after Ladozyme addinger. I have a samples for 80% and 60% solids. If sediment rolovap second sample for 60% solids. Use designated amount above (200gm —5 50gm for 80% solids. I sediment rolovap second sample to 60% solids. Use designated amount above (200gm —5 50gm for 80% and 150.38gm —5 50gm for 60%) - Label LAC-090209-A3-80/60, A6-80/60 and -A12-80/60 for 80% and if necessary 60% solids. I sespectively. DO NOT do Viscosity on 60 and 80% solids samples. Store 80% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week. Pre-weigh 1 filer Rolovap flask. Record weight of Rolovap flask. Record weight of Rolovap flask. Record weight of Rolovap flask. Asolids tests on sample from remaining "20% Solids" mixture. Do % Solids on sample from remaining "20% Solids" mixture. Do % Solids on sample from remaining "20% Solids" mixture. Add 1700 gm Di welter to glass reactor in water beth such that reaction mix lemp is 75C. Add 1700 gm Di welter to glass reactor in water beth such that reaction mix lemp is 75C. And 472 gm and red Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Mixe at 200 control. | | | | | | |
| and Notova pirst to Substance in the seadment, Stop and on ort following. It sedement rotovas second sample to 60% solids. Use designated amount above (200gm -> 50gm for 60% and 150.38gm -> 50gm for 60% and LAC-090209-A3-80/60, -A6-80/60 and -A1-280/60 for 60% and if necessary 60% solids, respectively. LAC-090209-A3-80/60, -A6-80/60 and -A1-280/60 for 60% and if necessary 60% solids, respectively. DO NOT do Viscosity on 60 and 80% solids samples Store 80% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week Pre-weigh 1 lifer Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids on sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Add 1700 gm Di water to glass reactor in water beth such that reaction mix temp is 75c Add 472 gm Di water to glass reactor in water beth such that reaction mix temp is 75c And 472 gm of the meate slowly to reactor while mixing until all Permeate is wetted and evenly suspended May a 720 for on an event page 100 for the page 100 | | At U, 3, 6, 9 and 12 rirs i | after Lactozyme addition - Take a samples for 80% and 60% Evap, grop pH to 4.0 with Cond HC. | _ | | |
| Starper to two 's solids' to 'see resignated amont above (2,00g) — 5 ogn not our's solids. The 's ogn not our's solids' to 'see resignated amont above (2,00g) — 1 ogn not our solids' to 'see a solids' to 'see a not our solids' to 'see a not our solids' to 'see a not our solids' samples in cold and take pics at 1 day and periodically over next week Pre-weigh 1 lifer Rotovap flask Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher 's solids tests Do % Solids tests Do % Solids to sample from remaining "20% Solids' mixture Do % Solids tests Add 1700 gm Di water to glass reactor in water bath such that reaction mix lemp is 75c Add 1700 gm Di water to glass reactor in water bath such that reaction mix lemp is 75c Add 425 gm died Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | and Rotovap lirst to 80% | % solids - if no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second | _ | | |
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| Record weight of Rotovap flask Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids on sample from remaining "20% Solids" mixture Do % Solids on sample from remaining "20% Solids" mixture Add 1700 gm DI water to glass reactor in water beth such that reaction mix temp is 75C Add 4720 gm died Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | L | Pre-weigh 1 liter Rr | Potestary John Sounds Samples in Cold and take product 1 day and ponedically of a most recent | No No No | + | 9 |
| Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids tests Do % Solids on sample from remaining "20% Solids" mixture 20% Permeate - 70C, 30 min - Then 0.2%Lactozyme3000 et 35C,pH6.5(w/HCl)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids Add 1700 gm Di water to glass reactor in water beth such that reaction mix temp is 75C Add 47S gm died Permeate slowly to reactor white mixing until all Permeate is wetted and evenly suspended | 1. | Record weight of R | Rotovap flask | | - | |
| % solids tests Do % Solids on sample from remaining "20% Solids" mixture 20% Permeate - 70C, 30 min - Then 0.2%Lactozyme3000 at 35C,pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids mi Add 1700 gm DI water to glass reactor in water bath such that reaction mix temp is 75C Add 41700 gm Di water to glass reactor in water bath such that reaction mix temp is 75C Add 44 CS gm divided Permeate stowly to reactor white mixing until all Permeate is wetted and evenly suspended | L_ | Transfer Calculated | ed gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher | | _ | |
| Do % Solids on sample from remaining "20% Solids" mixture 20% Permeate - 70G, 30 min - Then 0.2%Lactozyme3000 at 35C,pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids ml Add 1700 gm DI water to glass reactor in water bath such that reaction mix temp is 75C Add 4720 gm dede Permeate stowly to reactor while mixing until all Permeate is wetted and evenly suspended Add 425 gm dedee Permeate stowly to reactor while mixing until all Permeate is wetted and evenly suspended | | % solids tests | | | | |
| 20% Permeate - 70C, 30 min - Then 0.2%Lactozyme3000 at 35C,pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids ml Add 1700 gm Di water to glass reactor in water bath such that reaction mix temp is 75C Add 4725 gm died Permeate stowly to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 2nd Acad and acad and acad acad acad acad aca | Ш | Do % Solids on sar | Imple from remaining "20% Solids" mixture | | | |
| | 22 B | Permeate - 70C, 30 min - Then 0.2% | %Lactozyme3000 at 35C,pH6.5(w/HCl)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids | Ε | | Time |
| Add 425 gm dried Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | L | Add 1700 am DI water to | to class reactor in water bath such that reaction mix temo is 75C | | ł | |
| Mily at 700 favor prin | | Add 425 am dried Perme | leate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | | |
| | 1 | Mix at 70C for30 min | | | | |

Figure 10

| | Cool to 35C while mixing | | | |
|--|--|---------|--------------------|----------|
| | Adjust pH to 6.5 with NaOH or HCI while mixing | | | |
| | Record pH and control pH at 6.5 | | | |
| | Add 0.85 gm Lactozyme3000 to 50 ml water in beaker and mix well | | | |
| | Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing | | | |
| | At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - | | | |
| | 71 0,60,00,00,00,00 | | | |
| | At 0, 3, 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCI | | | |
| | and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second | | | |
| | sample to 60% solids. Use designated amount above (200gm> 50gm for 80% adn 150.38gm> 50gm for 60%) - Label | | | |
| | LAC-090209-B3-80/60, -B6-80/60, -B9-80/60 and -B12-80/60 for 80% and if necessary 60% solids, respectively. | | | |
| | | | | |
| | % solids samples in cold and take pics at 1 day and periodically over next week | t Flask | Wt Flask Target Wt | % Solids |
| | Pre-weigh 1 liter Rotovap flask | | | |
| | Record weight of Rotovap flask | | | |
| | Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher | | | |
| | % solids tests | | | |
| 3, 420 30 77 | Do % Solids on sample from remaining "20% Solids" mixture | | | |
| C 20% Permeate | 20% Permeate - 70C, 30 mln - Then 0.1%Lactozyme3000 at 35C,pH6.5(w/HCI)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids | E | 늄 | Tine |
| | Add 1700 gm DI water to glass reactor in water bath such that reaction mix temp is 75C | | | |
| | Add 425 gm dried Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | | |
| | Mix at 70C for30 min | | | |
| | Cool to 35C while mixing | | | |
| | Adjust pH to 6.5 with NaOH or HCI while mixing | _ | | |
| | Record pH and control pH at 6.5 | | | |
| | Add 0.43 gm Lactozyme3000 to 50 ml water in beaker and mix well | | | |
| | Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing | | | i |
| | At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - | | | |
| | LAC-390203-LU,C3,C9,C12 | Ī | | ľ |
| | At 0. 3. 6. 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap. drop pH to 4.0 with Conc HCI | | | |
| | and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second | | | |
| | sample to 60% solids. Use designated amount above (200gm> 50gm for 80% adn 150.38gm> 50gm for 60%) - Label | | | |
| | LAC-090209-C3-80/60, -C6-80/60, -C9-80/60 and -C12-80/60 for 80% and if necessary 60% solids, respectively. | | | - |
| | DO NOT do Viscosity on 60 and 80% solids samples | | | |
| | Store 80% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week WI F | t Flask | Wt Flask Target Wt | % Solids |
| | | | | |
| | Record weight of Rotovap flask | | | |
| | Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher | | | |
| | 70 Straids and from ramaining "2002, Califide" midura | Ť | | |
| The second secon | Ed in Sample nom lemaining 20 is boiled in the control of sample nome in the control of sample n | | | |

Figure 10 (Cont'd)

| % of Soy 3.00% 9 gm/Liter 12.75 g | |
|--------------------------------------|-------------------|
| % of Soy 3.00% 9 gm/Liter 12.75 g | g/mole |
| gm/Liter 12.75 g | % of Soy |
| | g Na2SO3/liter |
| Molar 0.1012 n | mole Na2SO3/liter |

| Г | | | _ | Г _{ь.} | 1 |
|------|--------------|----------|----------------|--------------------------|---|
| | 26.04 g/mole | % of Soy | g Na2SO3/liter | 0.0674 mole Na2SO3/liter | |
| , | 126.04 | 2.00% | 8.5 | 0.0674 | |
| 0000 | Na2SO3 | % of Say | gm/Liter | Molar | |

1,700.00 425.00 0.00 0.0000 0.0000 0.0000

Lactozyme3000

Water (gm) Permeate

Solids (pph) Solids (%)

Solids (pph) Solids (%)

| Water (gm) | 1,700.00 | |
|---------------|----------|--|
| Permeate | 425.00 | |
| | 0.00 | |
| Lactozyme3000 | 0.4250 | |
| | 0.000 | |
| | 0.000 | |
| | 0.00 | |
| Solids (pph) | 25.00 | |
| Solids (%) | 20.00% | |

Assumptions for LAC:
Lactozyme3000L = 3000 LAU/ml (1 LAU = 1 umale glucose/min) (Sigma price = \$129.50/250ml)
(PH6.5 / 37C / 4.5% w/w Lactose / 30 min / 0,035-0.1LAU/gm)

1 ml Lactozyme3000L -> 3x10³ umoles lactose * 1 mole lactose/10° umoles lactose * 342 gm lactose/mole lactose = 1.026 gm lactose/min

Therefore, for 1% enzyme loading (1 gm Lactozyme3000L/100 gm lactose) it will take approx 100 min to hydrolyze 100 gm lactose)

Figure 10 (Cont'd)

600.00

800.00

1200.00 400.00 800.00

| CAC-030203 | (20%Perm-0.5.0.2.0.1% Laccognis-3000-35C.pH6.5.12tws-HPLC,pxH4,Evac) |
|------------------------|--|
| 4.Parameters | \/ अंधरक |
| Lacrozyme3003L | itish oo Pemasaa sobaa |
| Ę | i& S-Controlled |
| Hydralysis Temp | 400 (Rx Mx Temp) |
| Latiose Source | :Ened Permeate |
| % Fermeste Solds | .20% |
| Crder Conder | Permeate-750, 1fr then 400. |
| pri Alter Enz Reaction | 4.0 w/Conc HCI then Retaining |
| œ | Vætsæs |
| 4.actoryree30000 | 10.2% on Permanente schödes |
| ā | & St. Controlled |
| 3-tydratysis Temp | 400 (Rx Mx Tenp) |
| Lactore Source | Ened Penneate |
| % Permeate Solids | |
| 0.00 | Peroxeste 7%C ffor their 40C |
| p+(Affer Enz Regotion | 4.0 wiCons HGI then Rotavep |
| Q | Vakors |
| Laoiczyms3000. | 10.1% on Fermiosis solicis |
| ě | i8.5-Commiled |
| 3-tydrofysis Temp | ACC (Fx Mx Temp) |
| Cactoge Source | Dried Permeate |
| % Permeate Solds | 20% |
| Order | Permeate-75C ftr frem 40C |
| phi Affer Enz Reaction | 4 0 wx2ono HCI then Rotevap |
| 524, 588, 590 | Vakors |
| 50.6 Sends | Redover cr. 10 % Soviets - Setting |
| 60A, 40B, 80C | Salvas |
| 80% Scrids | Potoxap is 86 % Sovieta. Setting |
| 784, 768, 70C | Valises |
| 70% Solds | Hotovap to 70 % Solide - Setting |
| 884, 8003, 80c | Values |
| | |

Figure 1

| Secretary of the process of the process of the pro- | | | | | | |
|---|------------------------------------|--------|-----------|------------|--------------|---|
| | (20% Perm-0.5,0.2,0.1% Lactoz) | nne300 | 0-35C,pH6 | .5,12hrs | HPLC,pH4,Eva | |
| 4 | i i | | Summer | o Ž | | |
| | | | | · · | | |
| ** 00000000000000000000000000000000000 | | : > | | | | |
| 0000000 | | * | | () | | |
| | | < | | | | |
| 24-202020247 | A PRODUCTION OF THE ACCORDANCE | æ : | | * : | | |
| | | Y | | × | | |
| 90008 | PoPerm 0.5%; acto300 | * | × | | × | - |
| 380209-A3- | PhPerm-0 5% Lacto300 | × | * | | × | |
| 380209-28 | **Perm-0.5%;3sta300 | * | × | | * | |
| LAC090209-As-80 | PoPerm-0.5%; secto300 | × | × | | × | |
| 980209-49 | %Perm-0.5%Lacto30 | × | × | | × | |
| 3802087 | ** Perm 0.5% (30000) | * | × | | × | |
| 20209-412- | PuPerm-0.5%; acto300 | × | × | | × | |
| 30209-A12- | 9% Perm-0 5% Lacto30% | × | × | | × | |
| 80000000 | MPerm 0.2%; 3330300 | × | | × | × | |
| - CACOSO209-B3 | 10.Ferm.C.2%;28Ct0308 | × | | * | | |
| 88-802080040 | M.Perm-0.2%:Lacto300 | × | | × | | |
| 88 6000600V | MPerm 0.3%; actood | * | | × | | |
| KC080208-B | 10.Perm 0.2% Lacto300 | × | | × | | |
| 25 CS-039 G02030777 | 60 20% Perm 0.2% Lacto3000-31/160% | ж. | × | | * | |
| 080208-83- | MPem-0.2%;.add300 | × | × | | * | |
| 98 502080 | %Perm 0.2%; acto300 | × | * | | × | |
| 090209-88 | M.Perm-0.2% (acto30) | × | × | | | |
| 080208-B& | 95Pemil: 2%; #dd330 | × | × | | * | |
| .090209-89 | %Perm-C.2%cacto300 | × | × | | × | |
| 90209-312 | %Perm-0.2%(assp30) | × | × | | | |
| 90209-312 | 90,Pemil 0.2%; 3820300 | × | × | | × | |
| *C080208- | %ജ്ഞ 5 % കൾഡ് | × | | * | | |
| C080208 | %Perm-0.1%Ladd30X | × | | × | | |
| | M.Pemil 1% Lacts300 | * | | × | | |
| 4C080208H | %Perm-C 1%LactodX | × | | * | | |
| 2090209-C | 9%Perm 0 1% (380030) | × | | × | | |
| 90209-03- | 16,Pem-0,1%; adic300 | * | × | | * | |
| LAC080208-C3-80 | %Perm-0-1%Lacco303 | × | × | | × | |
| 90209 C6 | %Perm 0.1% (acto30) | * | × | | × × | |
| 90208-08- | M.Perm-C. 1%!; addc306 | * | ж | | | |
| 90208-09 | M.Perm.C. (%Lacto30) | × | × | | × | |
| 90208 CS | %Perm 0.1%U3d030X | × | × | | × | |
| 0209-C12 | M.Perm.C. 1%: Acto.SR | × | × | | × | |
| 0209-C12 | 20%Perm 0.1%Lacto3000-12h-50% | × | * | | × | |

Figure 12

| Experiment Number: | Experiment Number: LAC-040208 |
|--------------------------|---|
| | [20%Perm.0.5,0.2.0.1% Lactozyme3000-35C,pH6.5,12hrs-HPLC,pH4,Evap) |
| Glucose Test Sample Prep | a displayed and a second a second and a second a second and a second a second and a second a second and a second a second and a second a second and |
| | 1 Phace 8 mt sample about in 16 mt centrituge tube |
| | 2] Add C 1 ml 10% 1803 to sample and mix (Enzyme kill step - should drop pH to 43. May have to adjust amount of acid) |
| 8 | 3 (Ditute unti) ma solidas are visible, no rifouctiness = clear solution |
| 4 | 4/Adjust DH to 44 with HCD if necessary |
| | 5[DO NOT FILTER, store in referigerator for HPLC analysis |

Figure 12 (cont.)

| AC-090209 | (20%Perm-0. | 5,0.2,0.1% | Laciozym | 3333 | (20%Perm-0.5, 0.2, 0.1% Lacinzyme 3000-350, pH6.5, 12hrs-HPLD, pH4, Evap) |
|------------------|-------------|--|----------|---------|---|
| LAC-090206 | % (actasss | 1 % solide1 % solide2 1 % solide3 jan id3 (AVG | %sofice? | %solo83 | solids(AVG) |
| LAC080209-40 | S | | | | *D(V)0 |
| LACCB0209-A3-60 | 250 | | | | W/\(\) C# |
| LAC050209-43-80 | 2.30 | | | | 8/XQ# |
| LAC090209-AS-80 | 930 | | | | (2)X(G |
| LAC090209-A6-80 | | | | | 820 |
| LAC030209-AS-80 | | | | | ig/AiG# |
| LAC090209-A9-80 | | | | | #O/V/@# |
| LAC380208-412-80 | | | | | #0MG# |
| LAC390209-A12-80 | | | | | in/inc |
| | | | | | *D/\0; |
| | | | | | #DIV/03 |
| LAC080209-80 | Œŝ | | | | iii/NC# |
| LAC090208-83-60 | | | | | 10/1C# |
| LAC090209-83-80 | | | | | \$0.00g |
| LAC030209-56-80 | | | | | #O/A/C# |
| LAC030209-B6-80 | 88 | | | | igy/iC# |
| LACUSUZUS-88-40 | | | | | #NVO! |
| LAC090209-BB-80 | 620 | | | | :0/AO# |
| LAC(902)9-8:2-60 | | | | | O/AC# |
| L#C090209-B12-80 | | | | | i0/xiC# |
| | | | | | io/Aig# |
| | | | | | io/Act# |
| UC090009-C0 | 0.10 | | | | 10/AC# |
| (AC098Z88-C3-60 | | | | | 10/40# |
| LACO88289 C3-80 | | | | | :0/AQ# |
| .AC090208-C6-60 | 610 | | | | #U/WG# |
| LAC090208-C6-30 | | | | | 10//iQ# |
| LAC0387208-09-50 | | | | | #DAG# |
| LACOS0209-C9-80 | | | | | 0/40# |
| LACOBORGE-C12-80 | 0.10 | | | | :0%C# |
| AC090289-C12-80 | | | | | io//io# |
| | | | | | 10 %0 |
| | _ | | | | 1 100 100 1 |

Figure 10

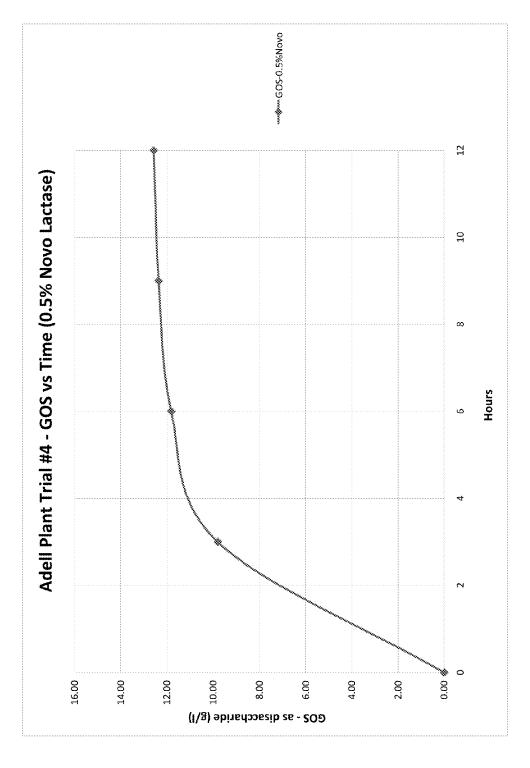
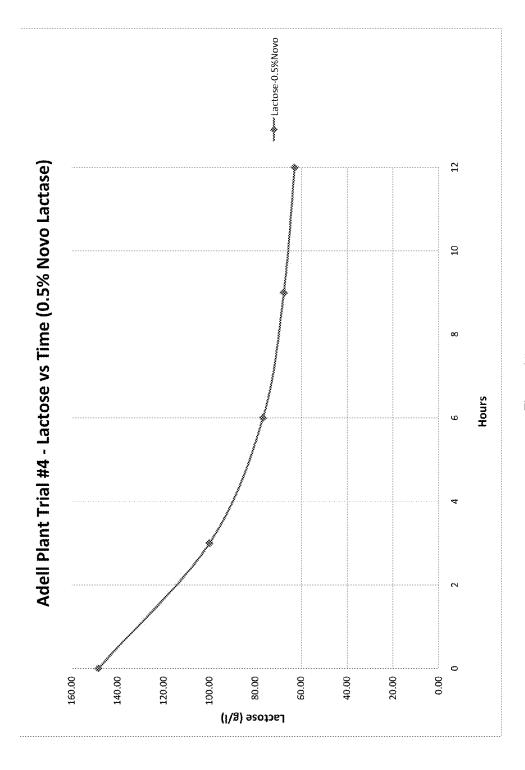


Figure 14



-igure 15

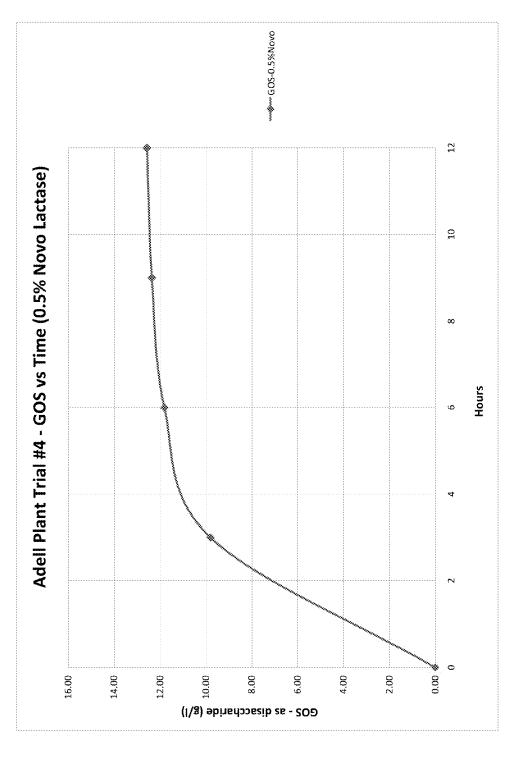


Figure 16

| MOVER L'ACTECLYTHE ACHION, COLL. | Cox. No. | C. C. CO. C. | | | ì | ١ | | |
|---|----------------|--|---|---|---|---|-----------|-------------------|
| | Lactose Source | 20% Репнева | 20% Penneade | 20% Perreads | 20% Permanee | 7 | Chacose | 28. 28. 28. |
| | Enzyme Level | 6.30% | 0.30% | 8388 | | 308.0 0 | | |
| | J.,., | 3 | | | 6 | 12 | Gabactuse | 185,16 |
| Lactore (+COS Disacchands) (artificial | Ł., | 28.8% | SS 33 | | 35.38 | 38.63 | | |
| | Ľ | 135.06 | 98.52 | | 88 | 28.82 | | |
| | SWK. | 133 70 | | | | | | |
| *************************************** | 200 | 190.061 | 80.17 | 48.00 | 88 86 | 28.36 | | |
| | CS | 87.6 | (e.e.g | NVXC* | 84.8 | 120 | | |
| | CER | 3.10% | 1.58% | 10/AC# | 3,627 | 8.73% | | |
| (2actose (9actos) | AVV. | 14694 | | 6983 () | 0.1007 | 0.0841 | | |
| (\$400000 (cast(\$)) | 158.1980 | 93.9 | 02.8¢ | 25.00 | 28.80 | K832 | | |
| *************************************** | 2nd neg | 0.04 | 808 | • | 13.85 58.51 | 48.95 | | |
| | · · | | *************************************** | *************************************** | ************************* | | | |
| | 3 | - | ~ | *************************************** | *************************************** | | | |
| | AVG | 3.57 | 80 OK | 58 60 | 13 10 10 10 10 10 10 10 10 10 10 10 10 10 | 200 | | |
| | GS. | 3.83 | 050 | #0000# | 080 | | | |
| *************************************** | RSD | • | 8≈ | CONON | 0.85% | 3250 | | |
| (Calcond second) | | 30143 | | 0.3765 | 0.3246 | ₹. | | |
| 1) Broad Recipionals | | 2.85 | 58 by | 23.00 | 888 | S8:90 | | |
| | 200 (60) | 8 | | | 56.22 | \$3.43 | | |
| | | | | | | | | |
| | | | | *************************************** | *************************************** | *************************************** | | |
| | 5A.C | 3.75 | 36 34 | 33.0% | 12.64 | 47.36 | | |
| | 28 | | 2000 2000 2000 2000 2000 2000 2000 200 | :::/\::O# | 0.50 | 77 | | |
| | 988 | XZ2./27 | :@/uC# | :0/A:0# | %803 3 | 0.93% | | |
| Calactore (molest) | AWS | 0.0208 | 0.3482 | 2500.0 | 0.3088 | 0.3817 | | |
| (f)(mp) #50/80000881(1-80%) | L | | 60 C) | 11.00 | \$2.0% | 6.03 | | |
| *************************************** | 200 000 | 300 | 19808 | | 10.07 | 85.58 | | |
| *************************************** | | | | • | , , , , , , , , , , , , , , , , , , , | | | |
| | **C | 000 | 28.15 | 28 | 6.17 | 8,15 | | |
| | as | 9000 | 1.34 | #08/0x | 5 (8) | E0.3 | | |
| | GSH | *000 | %9V ; ; | ic/Na* | (47% | 9,99,0 | | |
| GOS (Trisacchande-moles/I) | AVX3 | 0.05036 | 80.08 | 5.0021 | 2.0297 | 0.0008 | | |
| W. actore Hydrolyzed | *** | | 7003.1.7 | | 70.00% | 20028 | | |
| Control of the second | /isolati | | 20. X1.40 | | | | | |
| (hazed on (bloods) | (enriger) | | %89.65 % | 85.35% | %.C# 59 | 83.72% | | |
| Mass Balanco-n | | | - | *************************************** | *************************************** | | | |
| (Resed on Oknose Equivalents) | | | | | | | | |
| (assume trisacchande GOS) | (1000) | , | 300 80. | 38.44% | 4KO 32% | 82.50% | | |
| Rate of Lactode Hyperol | | | | | | | | |
| Affirmen Physicanomy | | | 380 | 38,50 | 2000 | 0.00 | | |

Figure 1

| Nave Lactorynae 30806. (ML) | Sample | Lo.C. 2699234-18 | 1.40,00000.4.8 | CAC-080324-8 | 5-900000-W1 | AC-080304-8 | Lessinge | 362.365 |
|--|----------------|---|---|--------------|--|---|---------------|---------|
| | 00000 000000 | 20% Psceneste | 20% Posmosic | 20% Personek | 20% Permests: | 20% Permedia K | Cotenose. | 280, 36 |
| | Erzyenk : 2008 | *08.8 | 2000 | ×2X.5 | 330% | 0.30% | | |
| | | G | · · | 60 | ê. | | Castroccoses: | 130,55 |
| Morros deblocados Signatores de la contraction d | 000 101 | 558.36 | 33 | 96.39 | 28.8% | 80.06 | | |
| | 200 (00) | 14.038 | 188 A | 68 33 | 88 | 3K.7K | | |
| | AVIS | 118.843 | *************************************** | | | | | |
| | SW | \$60,00 | 180 W | *** | 28.8% | 22.22 | | |
| | 2 | 0.86 | (2) | 6.13 | 8 | 20.0 | | |
| | RSD | 0.35% | 9,900 | 0.12% | X () | 0.03% | | |
| (((((((((((((((((((((((((((((((((((((((| 6305 | 19239 | 8.3377 | 13 182.0 | 84.2.0 | 0.5469 | | |
| Cacaoas socials | 16, 000 | 28.8 | 45.61 | 36.88 | 28.83 | 83.68 | | |
| | 200 102 | 8.72 | 48.60 | 30.75 | 38.80 | 19.64 | | |
| | - | | | | | | | |
| | 2 | | | | | | | |
| | S/W | 89.9 | 46.63 | 58.58 | 28.53 | \$5.63 | | |
| | 22 | 0.03 | 0.00 | 0.10 | 3.0 | 0.03 | | |
| | GSW | | 2000 | 3,35% | 6.18% | 0.05% | | |
| Salaran Charles Control | 405 | 0.00.0 | 12836.3 | 3X87 0 | 0.33308 | (3558) | | |
| Standord Control | 38, 690 | 11.72 | 42.67 | 48.83 | 25.87 | \$4.08 | | |
| | 200 300 | 188 14 | 42.87 | 203 (39 | 38.85 | 80.433 | | |
| | | - | - | | | | | |
| | * | | | , . | | | | |
| *************************************** | SWS. | 11.40 | 42.84 | ≫.S\$ | 28.86 | 80.67 | | |
| *************************************** | 3 | 2000 | 90.0 | 0.18 | 3.8: | 800 | | |
| | 928 | 2.34% | 35860 | 8.38% | *800 | 8 | | |
| Selectors (Selectors) | W.C | 0.0633 | 1,382.0 | 8228 | 0.3:06 | 3308 | | |
| San | | 300.0 | 12.05 | 346 | 14.03 4.03 | | | |
| | 2nd 1ep | 000 | 11.82 | 36.84 | ** | 14. £8 | | |
| | | | | | | | | |
| | 405 | 00.3 | 11.88 | ₹ | 30 31 | € | | |
| | 33 | 2003 | 82.3 | 200 | 80 | 8 | | |
| ********************************** | 388 | (0/AR)# | 2,5933 | 8.52% | *8000 | 2018 | | |
| GOS (Trasconarde notes) | COM | COXOC U | 09030 | 9880 3 | 0.70° | 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 | | |
| Mindeed hydrohaes | | | | _ | _ | | | |
| (bassed on Lactores) | (motas) | | 36. SW | %86 99 | 22.88% | 83.58% | | |
| Secure Hydrolyzed | | | | | , | | | |
| (based on Glomes) | (psych) | 7 | 3 | \$ 10% | X.73%; | 18/345 T. | | |
| Maco Salanca: % | | | | | | | | |
| decaped centifications Equivalence) | *** | | 454.4192 | (308 × 1) | 2001 045 | 110 46% | | |
| Security of the second control of the second | (0000) | *************************************** | | | Secretaria de la companya del companya del companya de la companya | | | |
| | | | 673 | 0.000 | 0000 | 2000 | | |
| | - | ······································ | | | | | | |

Figure 18

| Norm Lacsezyna 3000k. (BL) | Sample | CAC-0200204-C | U.C.0802384-C | CAC 080304-C | ~#63X80:3X | 24C090000. | 1.800000 | 342.33 |
|---|----------------|---------------|----------------|--|---|------------------|-----------|--------|
| | Cactose Soxers | 20% Permedie | 20% Pennessa | 20% Permade | 20% Permeate | 20% Permeste | Skuposo | 180.75 |
| ~~ | Fresyme Level | 3328 | 9.838 | 2333 | 0.30% | 2.30% | | |
| | 593 | 9 | ** | œ | œ | 1.2 | Gelectore | 180,18 |
| Lectose (+CCC Osacchardo) (smill) | 333 83 | 164.69 | 103.28 | 387 (S) | 89.27 | 76.37 | | |
| | 293 182 | 363.38 | 56.501 | 89.05 | 80 78 | 35.07 | | |
| | AVG | 165.02 | | _ | | | | |
| | 200 | 500.035 | 139:535 | \$6.57 | 33,73 | 78.8% | | |
| | CS | 633 | (6.9) | 898 | 0.50 | 3.42 | | |
| *************************************** | OS8 | 0.23% | %8) € | 0.77% | 2808 | 3986C | | |
| (Research association) | 2000 | 0.4674 | 6.3023 | 0.2587 | 39770 | *502'0 | | |
| (381086) | 187.986 | 503 | 05.85 | 43.75 | 98 79 | 28:3 | | |
| | Said ress | 8.36 | 15.35 15.35 | 44.26 | \$8.85 | 52,36 | | |
| *************************************** | | | | | | | | |
| | on. | | | ~ | | | | |
| | 388 | 50.8 | 12.88 | 28.82 | 40.27 | 37.50 | | |
| | 23 | Xc | 920 | 033 | 3.83 | 37.8 | | |
| | GS2 | 4 | 95200 | ************************************** | (%(%)) 2 | % ₹ % | | |
| (3)38(3) 82(3)(3) | 3/6 | \$223 | 8233 | 3.2444 | 0.3679 | 8,2862 | | |
| (3)(4)(3)(3)(3)(3)(3)(3)(3)(3)(3)(3)(3)(3)(3) | 9 | 19.5 | \$ X | (19.23 | (0) 94 | 48.87 | | |
| | | \$3.55 | 38.28 | 28.00 | *** 68) | 98.27 | | |
| | | | | - | *************************************** | | | |
| | | | | | *************************************** | | | |
| | OW | 13.83 | 38.23 | 45.88 | 48.36 | 20 0X | | |
| | S | හි | 8.07 | 3.40 | 3.44 | 98.0 | | |
| | ପଞ୍ଚଳ | 3,350 ° | 2, 19% | 0.93% | % % % % | 7.88.V | | |
| Galactose (moles/f) | 868 | C.0844 | 8.2424 | 0.5570 | 0.2374 | 0.2779 | | |
| SCS Trissoctiones (graft) | 18: N.S. | 0,00 | 83::5 | 12.55 | 13.63 | 12.68 | | |
| | (90) pt(; | 98 B | 27.5 | 22.03 | 92 ES | 34 505 14 505 | | |
| | | | | | | | | |
| | 500.00 | (C) (C) | *** ** | 22 | 13.69 | 13.96 | | |
| | 38 | 80.0 | 8.02 | 5.12 | (S) | 60°0 | | |
| | RSC | 320 | 3000 | 8000 | 0.33% | 223.0 | | |
| GOS (Trescripands-profesit) | XX | COGS II | 52822 | 59000 | 0.0000 | 3,040.0 | | |
| bezakorbek kanaber 18 | | | | | _ | | | |
| (tuned on Laybore) | (molar) | , | 35.37% | 44.85% | 47.67% | 35.84% | | |
| %: actose Hydrolyzed | | | | | | 1 | | |
| (hesed on Chicago) | (MCM) | | %.C9* | 9,000.74 | 27.32.8 | 61.06% | | |
| Wass Selance-% | | | | | ~~~ | | | |
| deased on Studose Egylesterials | incisti | , | 120,51% | 118,000 | 121,38% | 327,87% | | |
| Rate of Legisle - Kedos | | | | | | | | |
| g@trote@trakesard{ | | | 6.254 | 2000 | CONCE | 9.073 | | |

Figure 19

LAC-090209A-0.5% Novo, pH6.5

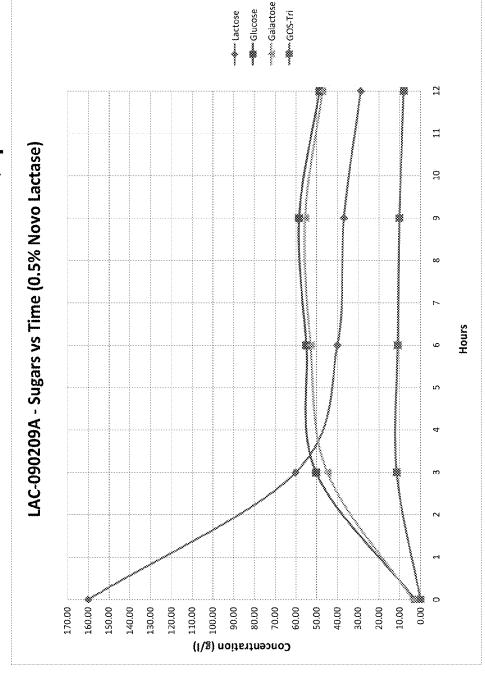


Figure 20

LAC-090209B-0.2% Novo, pH6.5

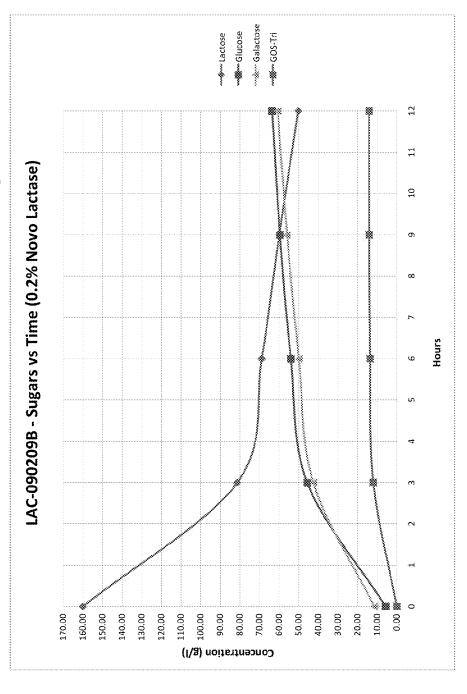


Figure 21

LAC-090209C-0.1% Novo, pH6.5

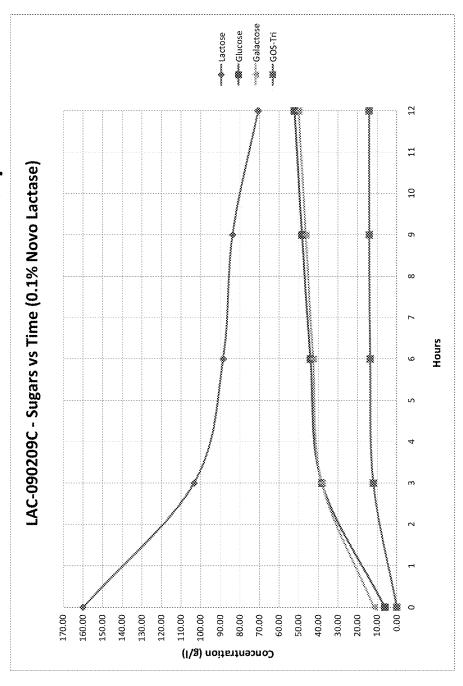


Figure 22

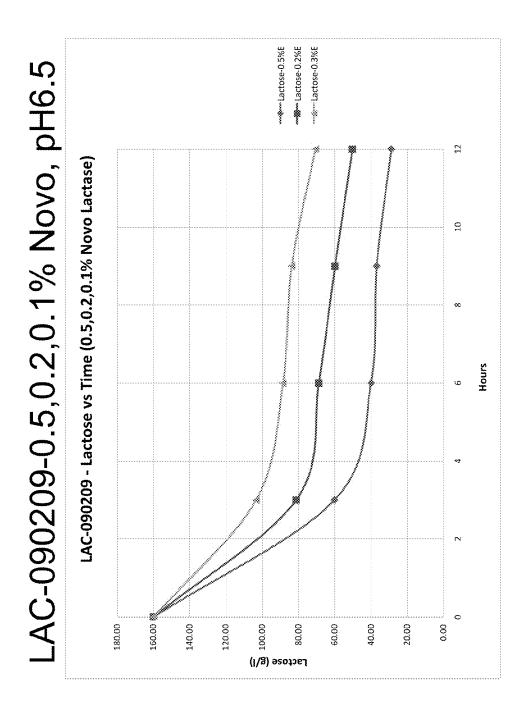


Figure 23

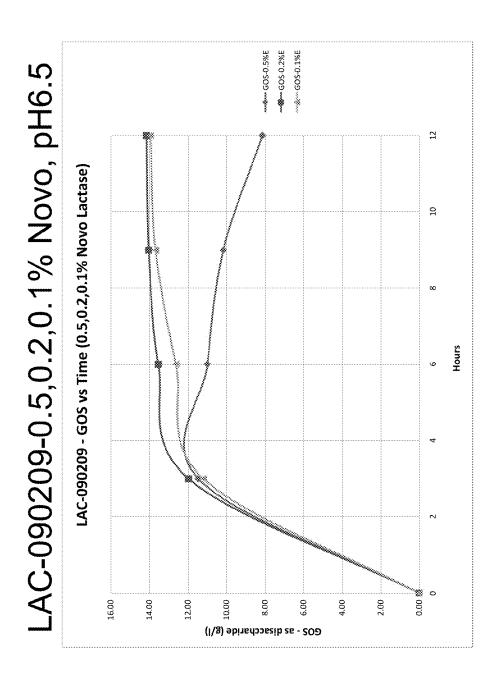


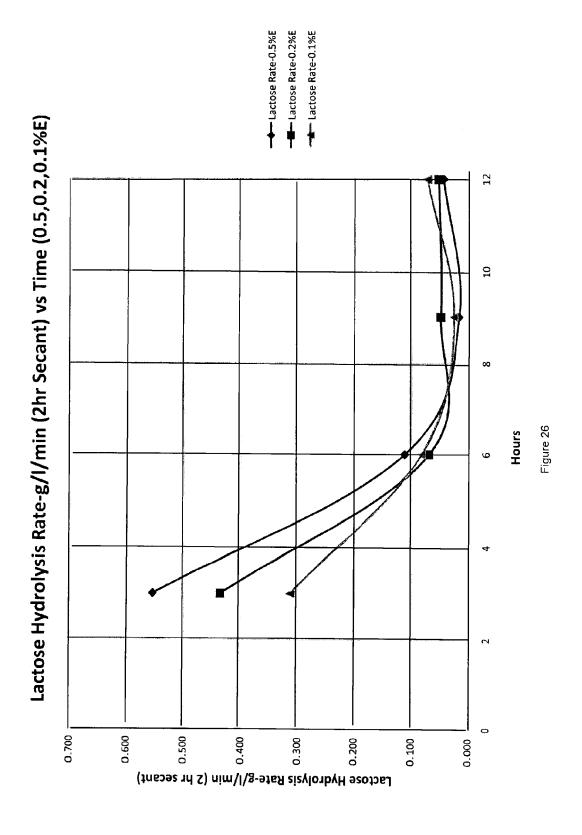
Figure 24

| A 1700.0 A 1700.0 A 25.0 A 25.0 A 25.0 A 25.0 A 25.0 A 20.0 B 1700.0 B 1700.0 A 20.0 B 1700.0 | NUMB | it Number: LAC-090209 | [(20%Perm-0.5,0.2,0.1% Lactozyme3000-35C,pH6.5,12hrs-HPLC,pH4,Evap) | | | |
|--|---------|-------------------------|--|---------------|-----------------|----------|
| To determine the effect of hydrolysis of 20% Perm with Novo Laciozymedion at 0.5, 0.2 and 0.1% on Permeate Soulds, at 305, pHS 5 to 12ths, followed by adjusting pH to 4.0 with conc. HCL fellowed by engocration to 60.6, 80%, solidis. 4.25.0 pm Deed Permeate 6.26.0 pm | Referen | | | | | |
| 1700 gan Water 2.13 gan Divide Demosts 2.23 gan Divide Demosts 2.23 gan Divide Demosts 2.23 gan Divide Demosts 2.25 gan Divide Demosts | | | To determine the effect of hydrolysis of 20% Perm with Novo Lactozyme3000 at 0.5, 0.2 and 0.1% on Permeate Solids, at 35C, pH6.5 for 12hrs, followed by adjusting pH to 4.0 with cone HCl, followed by evenoration to 60 & 80% solids. | | | |
| 4250 pm Diei Permetale 2.13 pm Neou Legicoryme 300001. | als: | |) gm Water | 11 | | |
| 2.2.13 min Nove Lactocyme 3000). 1700 grm Widers 4.55 0 min Died Permeate 4.55 0 min Died Semeste 5.50 0 min There Semeste 5.50 0 | | 425.0 | gm Dried Permeate | 1 | | |
| 1200 gm Under Permente 1200 gm Under Perme | | 2.13 | gm Novo Lactozyme 3000L | | | |
| 1.00 gray Vivient | | | gm Water | | | |
| 1700.0 gm Water 1.000 20 Min Header 2.000 2. | | 425.0 | gm Dried Permeate | | | |
| 1700 gpm Water 1700 | | 0.85 | Jgm DFL5000 | | | |
| 0.45 grap Die Die Permate | | |)gm Water | | | |
| 0.43 pin DFL5000 120 pto 500 Solids Evep 126 500 Solids Evep 127 Solids Evep 127 Solids Evep 128 pin 0.13 gam Evep 128 pin 0 | | 425.0 | gm Dried Permeate | _ | | |
| Evap 125 gm Evap 126 gm to 140 gm Evap 126 gm to 143 gm Evap 126 gm to 150 gm to 142 gm to 145 gm Evap 126 gm to 150 gm to 142 gm to 145 gm Evap 126 gm to 150 gm to 142 gm to 145 gm Evap 126 gm to 150 gm to 142 gm to 145 gm Evap 126 gm to 150 gm | | 0.43 | lgm DFL5000 | | | |
| 125 to 50 gm Evap 112 gm to 140 gm Evap 120 gm to 140 gm Evap 120 gm to 140 gm Evap 120 gm to 130 gm Evap 120 gm to 130 gm Evap 120 gm to 130 | | | Evap to 50% Solids see a second secon | (20%)*(125) | =(50%)*X | |
| Evep to 60% Solids Evep 160% | | | Evap 125 gm to 140 gm | X=(20)*(125) | ⊬(20)≍ | - 20 |
| 150.38 to 50 gm Evep 1054.80 gm to 133 gm Evep 1054.80 gm Evep 1056.80 gm Evep | | | Evap to 60% Solids | (20%)*(150 | *(%09)=(86 | × |
| Every 10, 20% Solids Every 10, 20% Every 10, 20% Solids Every 10, 20% Ev | | 150.38 to 50 gm | Evap 150.38 gm to 133 gm | X=(20)*(150 | 38)/(60)= | |
| 174.63 to 50 gm Evap 174.63 gm to 136 gm Evap 200 gw Solids Evap 200 gm to 50 gm Evap 200 gw Solids Evap 200 gm to 50 gm Evap 200 gm to 65% Lactocyme3000 at 35C, pH6.54wHCl). Then pH4.0(wHCL), Evap to 60 & 50 % Solids ml pH Evap 200 gm to 10 water to glass reactor in water beth such their reaction mix temp is 75C Add 40 20 gm to 10 water to glass reactor while mixing until all Permeate is wated and evenly suspended mlx at 70C for30 min Then 65% Lactocyme3000 to 20% Permeate at 35C while mixing Add 40 20 gm to 40 gm to 40 gm Water in beaker and mix well Record pH and control pH at 8.6 Record pH and 20 m | | | Evap to 70% Solids as a second | (20%)*(174 6 | *(%07)=(85 | × |
| Evap to 80% Solids Evap to | | 174.63 to 50 gm | Evap 174.63 gm to 136 gm [11] 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 | X=(20)*(174 | 63)/(70)= | |
| rep Tab) - | | | Evap to 80% Solids on appearance and a second secon | (100%)*(2001) | -(80%)*X | |
| rep Tab) - rep Target Wt | | | EVAP 200 gm to 60 gm to 20 gm | X=(20)*(200) | ((80) ((80)= | 8 |
| rep Tab) - noc HCi onc HCi sek WA Flask Target Wt or higher mi pH | | A 20% Permeate - 7 | 70C. 30 min - Then 0.5% Lactozyme3000 at 35C. pH6.5/w/HCh-Then pH4.0/w/HCl.). Evan to 60 & 80%. Solide | E | 1 | Į. |
| rep Tab) - onc HCi on data 6) - Label or higher or higher | | | Add 1700 gm Di water to glass reactor in water beth such that reaction mix temp is 75C | | 1 | 2 |
| rep Tab) - onc HCi ond (s) - Label cor higher mi pH | | | Add 425 am dried Permeate slowly to reactor while mixing until all Permeate is wetted and eventy suscended | | | |
| onc HCi ond HCi ond higher or higher mi pH | | | Mix at 70C for 30 min | | | |
| rep Tab) - onc HCi ond 6) - Label ek Wr Flask Target Wr or higher mil pH | | | Cool to 35C while mixing | | | |
| rep Tab) - onc HCI ond 6) - Label or higher or higher ml pH | | į | Adjust pH to 6.5 with NaOH or HCl while mixing | | | |
| rep Tab) - onc HCi ond (s) - Label cor higher or higher | | | Record pH and control pH at 6.5 | | | |
| onc HCi ond (a) - Lebel (b) - Lebel or higher mil pH | | | Add 2.13 gm Lactozyme3000 to 50 ml water in beaker and mix well | | | |
| rep Tab) - onc HCi ond 6) - Label ek WA Flask Target WA or higher mil pH | | | Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing | | | |
| ond HCI ond (s) - Label (s) - Label or higher or higher ml pH | | | At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) | | | |
| ond HCI ond 6) - Label cor higher ml pH | | | LAC-090203-A0,A3,A6,A12 | | | |
| ond 6) - Label of higher ml pH | | | At 0. 3. 6. 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evan drop pH to 4.0 with Conc HCI | | | |
| or higher ml pH | | | and Rotovap first to 80% solids - if no sediment. Stop and do not rotovap to 60% solids. If sediment rotovap second | | | |
| or higher ml pH | | | sample to 60% solids. Use designated amount above (200gm -> 50gm for 80% adn 150.38gm -> 50gm for 60%) - Label | - | | |
| or higher ml pH | | | LAC-090209-A3-80/60, -A6-80/60, -A9-80/60 and -A12-80/60 for 80% and if necessary 60% solids, respectively. | | | |
| or higher | | | DO NOT do Viscosity on 60 and 80% solids samples | | Н | |
| or higher ml pH | | | Store 80% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week | | \dashv | % Solids |
| or higher ml pH | | | Pre-weigh 1 liter Rotovap flask | | | |
| or higher ml pH | | | Record weight of Rotovap flask | | | |
| Ha le | | | Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher % solids fests | | | |
| Hd | | | Do % Solids on sample from remaining "20% Solids" mixture | | | |
| | | B 20% Permeate - 70 | 70C. 30 min - Then 0.2% actosyme3000 at 35C pH6 5(W/HC). Then pH4 0(W/HC)). Evan to 60 & 80% Solids | Ē | 7 | Time |
| Add 455 gradied Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended Mix at 700 finction mix | | | Add 1700 cm Di water to class reacher in water both each that reaching mix famule 750 | | | |
| Mix at 70°C for50 min | | | <u>revo. 1700 tim tim waren to grass treatons in waren barin stori intal reacuoir mix temps is 700.</u> 1704 1700 tim tim waren to grass treatons in waren barin stori intal reacuoir mix temps is 700. | | | |
| | | | Mix at 70C for 30 min | | | |

igure 25-

| | A distant and a second | | | |
|----------------|--|----------|--------------------|----------|
| | Aginst physocial Nach of The Wille Highligh | | | |
| | Record pH and control pH at 6.5 | | | |
| | Add 0.85 gm Lactozyme3000 to 50 ml water in beaker and mix well | | | |
| | Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing | | | |
| | At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - | | | |
| | LAC-090209-B0,B3,B6,B9,B12 | | | |
| | | | | |
| - | At 0, 3, 6, 9 and 12 hrs after Lactozyme addition - Take a samples for 80% and 60% Evap, drop pH to 4.0 with Conc HCI | | | |
| | and Rotovap first to 80% solids - If no sediment, Stop and do not rotovap to 60% solids. If sediment rotovap second | | | |
| | sample to 60% solids. Use designated amount above (200gm> 50gm for 80% adn 150.38gm> 50gm for 60%) - Label | | | |
| | LAC-090209-B3-80/60, -B6-80/60, -B9-80/60 and -B12-80/60 for 80% and if necessary 60% solids, respectively. | | | |
| | DO NOT do Viscosity on 60 and 80% solids samples | | | |
| | Store 80% and if necessary 60% solids samples in cold and take pics at 1 day and periodically over next week | Wt Flask | Wt Flask Target Wt | % Solids |
| | Pre-weigh 1 liler Rotovap flask | | | |
| | Record weight of Rotovap flask | | | |
| | Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher | | | |
| | % solids tests | | | |
| | Do % Solids on sample from remaining "20% Solids" mixture | | | |
| C 20% Permeate | 20% Permeate - 70C, 30 min - Then D 1%Lactozyme3000 at 35C,pH6.5(w/HCl)-Then pH4.0(w/HCL), Evap to 60 & 80% Solids | JE. | 듐 | Time |
| | Add 1700 gm DI water to glass reactor in water bath such that reaction mix temp is 75C | | | |
| | Add 425 gm dried Permeate slowly to reactor while mixing until all Permeate is wetted and evenly suspended | | | |
| | Mix at 70C for30 min | | | |
| | Cool to 35C while mixing | | | |
| | Adjust pH to 6.5 with NaOH or HCl while mixing | | | |
| | Record pH and control pH at 6.5 | | | |
| | Add 0.43 gm Lactozyme3000 to 50 ml water in beaker and mix well | | | - |
| | Add well mixed Lactozyme3000 to 20% Permeate at 35C while mixing | | | |
| | At 0, 3, 6, 9 and 12 hrs after Lactozyme3000 addition sample for HPLC Sugar Assays (see Sugar Test Sample Prep Tab) - | | | |
| | LAC-090209-C0, C3, C6, C9, C12 | | | |
| | Other Care Hand of the color of | | | |
| | A Copyright for the State of Copyright of of Co | | | |
| | cannot store at 80% soldier The administration of the control of the soldier. The soldier increase account control of 80% soldier The administration of the soldier of the | | | |
| | sample to one soules. Ose designated amount acrost except to soules and resource to one of the control of the c | | | |
| | DO NOT do Viscosity on 60 and 80% solids samples | | | |
| | ld and take pics at 1 day and periodically over next week | Wt Flask | Wt Flask Target Wt | % Solids |
| | | | | |
| | Record weight of Rotovap flask | | | |
| | Transfer Calculated gm (see above) into 1 liter Rotovap flask and refrigerate remainder of reaction mixture for higher | | | |
| | % solids tests | | | |
| | Do % Solids on sample from remaining "20% Solids" mixture | 1 | | 200000 |
| | | | | |

Figure 25 (Cont'd)



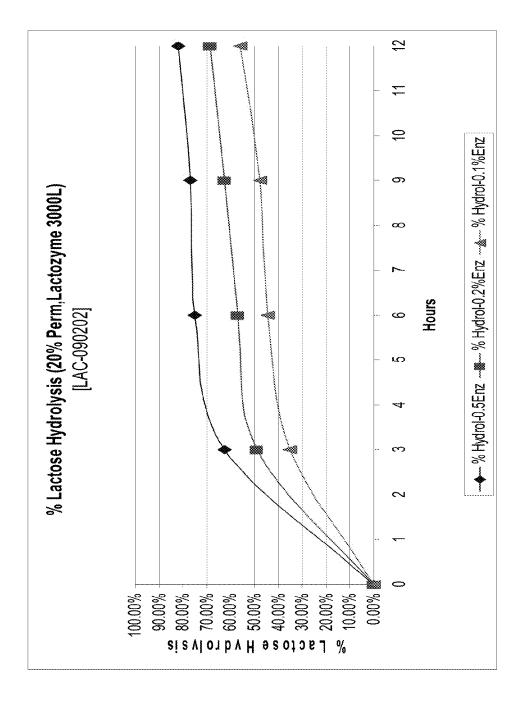


Figure 27

| MONO LACKSCHIME AURISE (M.L.) | Service | 2 | S & & | 0.50 | 0.800 CW | ٠ ٥. |
|--|--------------------|--------------|---|--|--------------|----------------|
| | (1,000000 Notions) | 16% Perceate | TON Percent | 10% Periodate | 18% Permesse | 197K Permerake |
| | . Kop otte | ×××× | 9000 | 2000 | **** | 0.350 |
| | £ | c | | * | 30 | :2 |
| (2000-00-00-00-00-00-00-00-00-00-00-00-00 | 100.000 | 23.854 | *** | | | 200 |
| | Sections | 148.80 | 2000 | 15 (A) | 31.43 | 8 |
| | 2000 | 3.8.6% | | | | |
| | 34.4 | > 00 | ** | 330 | 200.00 | ** |
| | **** | | 3 | ě. | *** | ** |
| | 088 | \$45.0 1 | 3338 | 388 | . 883 | \$20.0 |
| \$9000 account | 200 | 0.000 | 0.000 | 35.3.2 | 18/6/3 | 3000 |
| 0.0013600000000 | 3 | 33 | 8 | 33.00 | 300.00 | % S9 |
| | 200,000 | 22.2 | % & | 98 | 28.07 | 38.85 |
| 中央中央企业中央中央中央中央市场中央中央中央中央中央中央市场中央中央市场中央市场中央市场市场市场市场 | | | | | | |
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| *************************************** | 2000 | *** | ×82 | *** | X3.80 | 88 SS |
| | 68 | 88 | 800 | CCC | 800 | 800 |
| | OS X | | 288% | 0.00% | 4.388 | * ::00 |
| 00000 000000 | 202 | 2000 | 3,1683; | 1668 | 28.82 | 2388 |
| 667,866,0869 | 18. XX | 30 | ** ** | 100 00 100 00 10 | 2000 | 88 |
| | ***** | × × | 8 | | 28.8 | 8 |
| | | | | - | | |
| | \$1 | 28.2 | 250.00 | 236.35 | | 38.95 |
| | | | | - AXX | | 8/3/ |
| | 000 | 2000 | | 10000 | 200.3 | |
| | | 2000 | 8074.3 | 1000 | 2000 | |
| | | | *************************************** | 43333 | | 92.53 |
| | | | 322 | 438.52 | | 58.83 |
| | 80,00 | W.X | 000 | 388.7 | \$ XX | 20.00 |
| | X.03 | 200 | 880 | 1881 | 9K (3 | 86.2 |
| | | | | | | |
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| (のこうできた)をおりのあるとなったこのできた。 | (0.00.0) | | Č. | 8 | y | 8000 |

% Lactose Hydrolysis Using Validase (0.025 - 0.5 %Erz, Permeate Substrate)

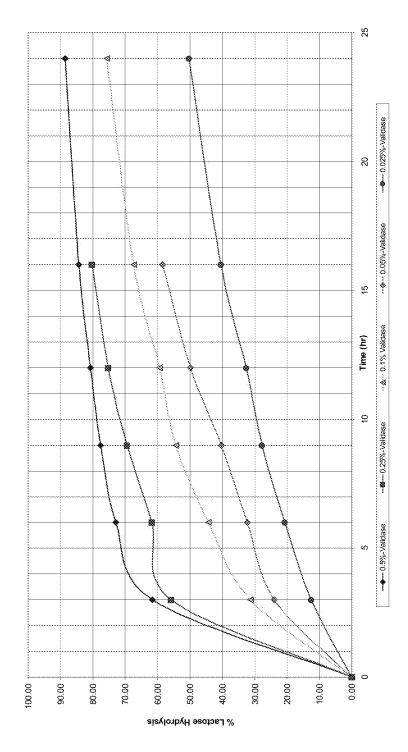


Figure 29

Enzymatic Hydrolysis Using Validase (0.50 gE / 100g Lac Loading, Permeate Substrate)

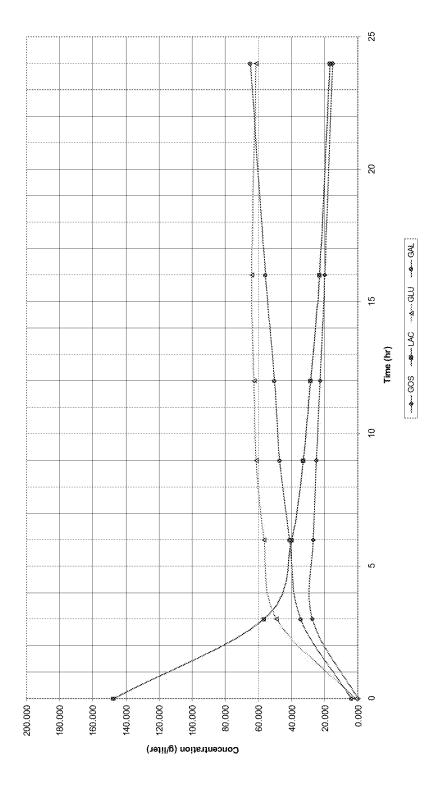


Figure 30

Enzymatic Hydrolysis Using Validase (0.25 gE / 100g Lac Loading, Permeate Substrate)

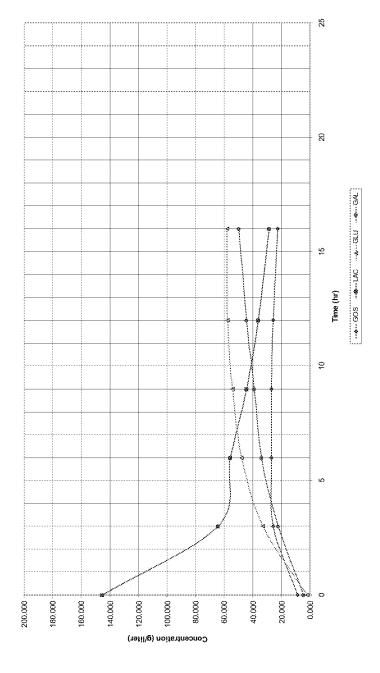


Figure 31

Enzymatic Hydrolysis Using Validase (0.10 gE / 100g Lac Loading, Permeate Substrate)

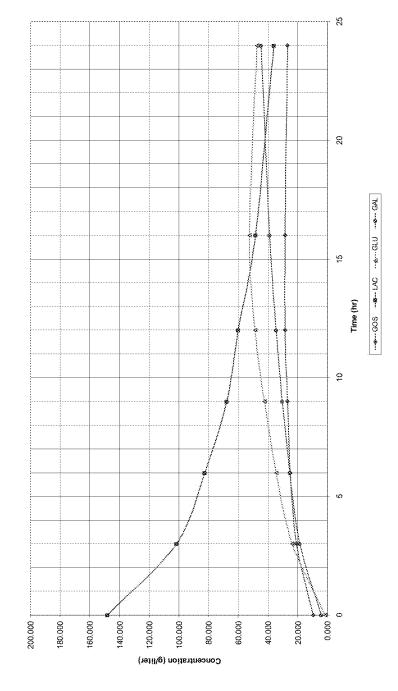


Figure 32

Enzymatic Hydrofysis Using Validase (0.05 gE / 100g Lac Loading, Permeate Substrate)

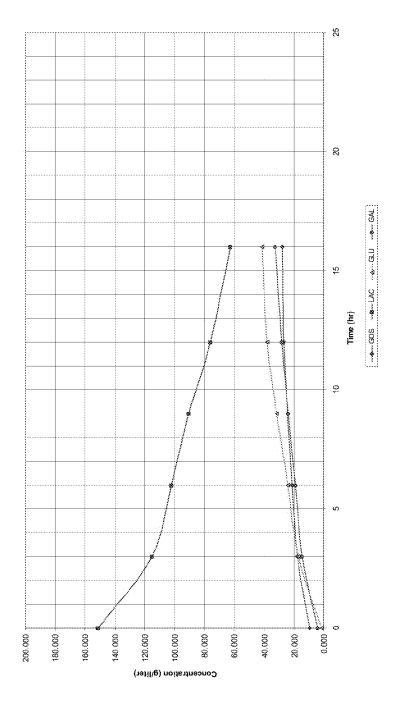


Figure 33

Enzymatic Hydrolysis Using Validase (0.025 gE / 100g Lac Loading, Permeate Substrate)

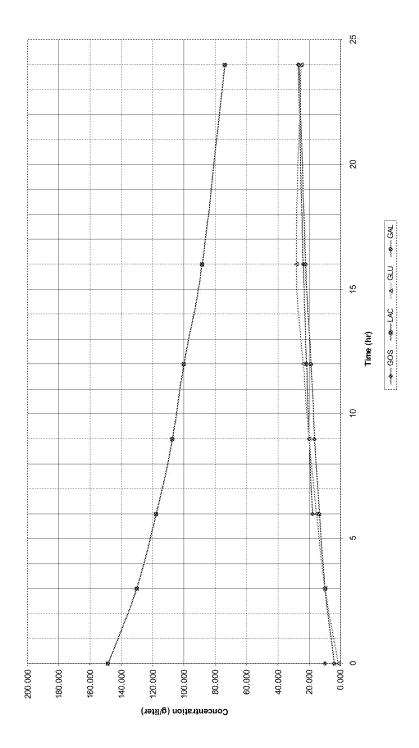


Figure 34

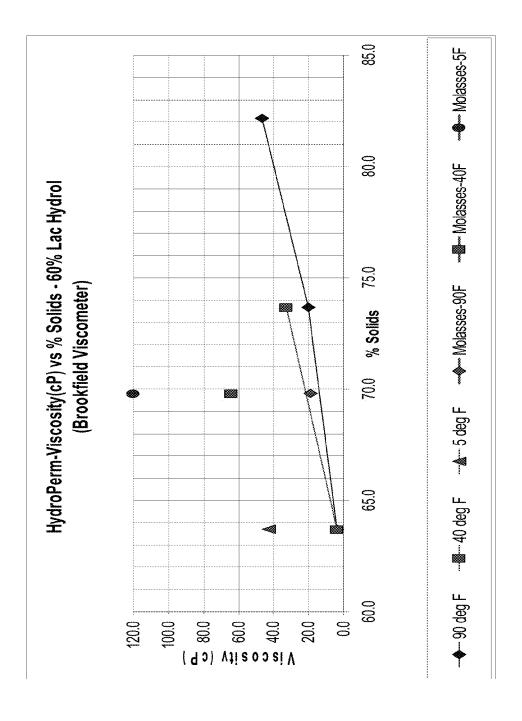


Figure 35

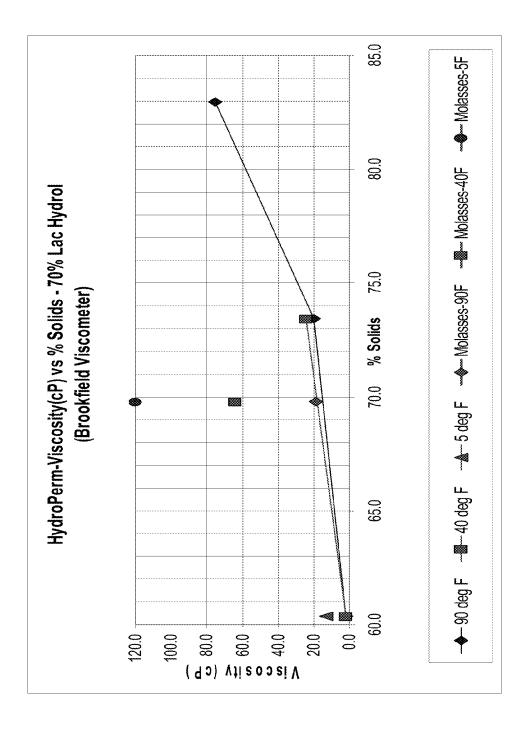


Figure 36

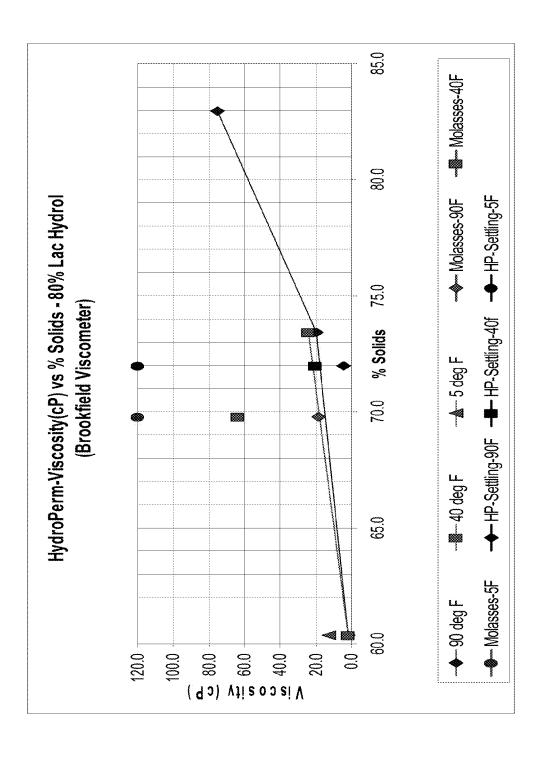


Figure 37

| Sample # | Hydrol | % Solids | 90F | 40F | 5F |
|---------------------|---------|-------------|------|------|-------|
| Тетр | Estim % | % | 06 | 40 | 5 |
| LAC-100208-A3hr-60 | 60 | 63.7 | 3.5 | 3.8 | 43.0 |
| LAC-100208-A3hr-70 | 60 | 73.7 | 20.2 | 32.9 | |
| LAC-100208-A3hr-80 | 09 | 82.2 | 46.6 | | |
| LAC-100208-B6hr-60 | 70 | 60.4 | 2.1 | 2.0 | 12.9 |
| LAC-100208-B6hr-70 | 70 | 73.4 | 20.1 | 24.0 | |
| LAC-100208-B6hr-80 | 70 | 83.0 | 75.4 | | |
| LAC-100208-C12hr-60 | 80 | 63.6 | 3.3 | 3.2 | 17.8 |
| LAC-100208-C12hr-70 | 80 | 70.0 | | 18.0 | |
| LAC-100208-C12hr-80 | 80 | 79.1 | 14.4 | | |
| Molasses | | 69.8 | 18.7 | 64.1 | 120.0 |
| HP-Settling Sample | | 72.0 | 4.2 | 20.2 | 120.0 |

Figure 38

NON-SETTLING HYDROLYZED WHEY PERMEATE CONCENTRATE AND RELATED METHODS AND NUTRITIONAL COMPOSITIONS

RELATED APPLICATION DATA

This application is a continuation of U.S. patent application Ser. No. 12/729,055, filed Mar. 22, 2010, which claims the priority benefit of U.S. Provisional Patent Application Ser. Nos. 61/162,164, filed Mar. 20, 2009, and 61/162,178, filed Mar. 20, 2009, which are hereby incorporated herein in their entirety by reference.

TECHNICAL FIELD

The present invention relates to whey permeate hydrolysate concentrates for nutritional supplement compositions and foodstuffs that may be used for livestock and humans, and methods of their production and use.

BACKGROUND OF THE INVENTION

Whey comes from the manufacture of cheese. Whey 25 permeate (sometimes also referred to as permeate processed whey) comes from a process of removing at least some of the protein from whey. The whey permeate is usually condensed to remove at least some of the water. A typical condensed whey permeate comprises about 35-45% by 30 weight total solids, of which total solids about 75-80% by weight is lactose.

It is beneficial to produce whey permeate hydrolysate concentrates for use in a wide variety of animal and human foodstuffs and nutritional compositions and supplements. 35 These uses include use of whey permeate hydrolysate concentrates as substitutes for other components of foodstuffs and nutritional compositions.

One of the difficulties in the transport and use of whey permeate hydrolysate concentrates is that the liquid products 40 are subject to sedimentation such that they cannot perform as desirable in a wide variety of industrial and agricultural uses that may require storage or transport over time and temperature profiles where sedimentation occurs, thus preventing their benefits from being realized.

In addition, liquid whey permeate hydrolysate concentrates also may be of such high viscosity that they can be unsuitable for industrial and agricultural uses that involve pumping, conduit transport or pouring.

Accordingly, where it is desired to use liquid whey 50 permeate hydrolysate concentrates in industrial food production, such as in the production of animal feeds or food preparation processes, it is beneficial to be able to provide liquid whey permeate hydrolysate concentrates that have beneficial solids contents, while maintaining sufficiently 55 good viscosity and resistance to sedimentation that they may be pumped, transferred by conduit or poured in industrial settings.

Accordingly, there remains a need for whey permeate hydrolysate concentrates that offer all of the nutritional 60 benefits of hydrolyzed lactose and resultant galactooligosaccharides (GOS), but likewise offer an advantageous collection of concomitant physical properties, such as high solids, as well as suitable viscosity and resistance to sedimentation over typical storage periods and within temperature ranges 65 experienced in various storage and transportation conditions, and in application climates.

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SUMMARY OF THE INVENTION

The present invention includes a method for producing non-settling hydrolyzed whey permeate concentrate, the hydrolyzed whey permeate concentrate produced thereby, and nutritive products containing same or produced therefrom.

The present invention also includes nutritional supplement and additive compositions in liquid or solid form that may be used in a wide variety of human and livestock applications and the like, such as for livestock feed mixture. Also included are methods of preparing the nutritional supplement and additive compositions, as well foodstuffs, using hydrolyzed whey permeates made in accordance with the present invention.

The present invention includes hydrolyzed whey permeate concentrates that may be rendered into a solid form, and which as liquids exhibit improved bulk handling and storage properties, as well as improved flow and non-settling properties, and which can be processed through liquid handling equipment, such as through pumps, conduits and the like.

The characteristics of the non-settling hydrolyzed whey permeate (NHWP) prepared in accordance with this embodiment of the present invention include the ability to concentrate a hydrolyzed whey permeate to a pumpable, pourable, non-settling liquid, most preferably with 75-80% solids.

As to the non-settling parameters of the liquid composition of the present invention, these compositions have a sedimentation rate such that the liquid product may be held at 90 F for at least two weeks with no appreciable sedimentation, preferably up to four weeks and beyond.

Method of Producing a Non-Settling Hydrolyzed Whey Permeate

The present invention includes a method of producing a non-settling hydrolyzed whey permeate with an enzyme, the method comprising the steps: (a) subjecting the whey permeate having an initial solids content in the range of from about 15 to about 25 percent solids to hydrolysis by an enzyme, so as to obtain a whey permeate hydrolysate having a degree of hydrolysis above about 65 percent and preferably between about 65 percent and about 80 percent, and (b) subjecting the whey permeate hydrolysate to evaporation so as to bring the level of solids in the whey permeate hydrolysate to within a range of from about 60 to about 80 percent solids, so as to obtain a whey permeate hydrolysate concentrate whose settling profile is such that there is no detectable settling over two weeks when stored at 90 degrees F.°, which corresponds to a pellet volume of approx 0.2-0.4 ml as measured in the specified Centrifuge Settling Test.

The parameters of the Centrifuge Settling Test are as follows:

- 1. Table top centrifuge
- 2. 1,250 RPM
- 3. Radius 14 cm.
- 4. Calculated G force: 244 g
- 5. Test temperature 75 F
- 6. Pellet volume measured vs. time.

The present invention include a method of producing a non-settling hydrolyzed whey permeate with an enzyme, the method comprising the steps: (a) maintaining whey permeate having an initial solids content in the range of from about 15 to about 25 percent solids in a reaction vessel at a temperature in the range of from about 70° to about 80° C. for a period in the range of from about 15 to about 45 minutes; (b) cooling the whey permeate to within a temperature range at which the enzyme is reactive; (c) subjecting the whey permeate to hydrolysis by the enzyme at a

temperature in the range of from about 25° to about 50° C. and at a pH in the range of from about 5.0 to about 7.5 for a period of time in the range of from about 3 to about 24 hours, so as to obtain a whey permeate hydrolysate; (i.e., the reaction mixture need only be in the pH and temperature range of any chosen lactase enzyme—e.g. fungal could be as low as pH3, some thermophylic lactases (not yet commercially available) could be as high as 60° C.); (d) subjecting the whey permeate hydrolysate to evaporation so as to bring the level of solids in the whey permeate hydrolysate to within a range of from about 60 to about 80 percent solids, so as to obtain a whey permeate hydrolysate whose settling profile is such that there is no detectable settling over two weeks when stored at 90 deg F.°, which corresponds to a 15 pellet volume of approx 0.2-0.4 ml as measured in the specified Centrifuge Settling Test.

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It is preferred that the whey permeate has an initial solids content in the range of from about 18 to about 20 percent solids. The preferred enzyme concentration is in the range of 20 from about 0.08%-0.12% of the reaction mixture for this solids range.

It is also preferred that the hydrolysis by the enzyme is carried out at a temperature in the range of a temperature in the range of from about 25° to about 50° C. and at a pH in 25 the range of from about 5.0 to about 7.5 for a period of time in the range of from about 3 to about 24 hours, most preferably from about 35° to about 40° C., and that the hydrolysis by the enzyme is carried out at a pH in the range of from about 6.5 to about 7.0.

The hydrolysis reaction typically will be carried out for a period of time in the range of from about 4 to about 16 hours, preferably 12-16 hours. Typically, the degree of hydrolysis will be above about 65 percent, and preferably in the range of from about 65 percent to about 80 percent. In addition, 35 under the prescribed enzymatic concentration, the liquid whey permeate hydrolysate condensate will have a galactooligosaccharides (GOS) content in the range of about 3% to 5% by weight.

The evaporation step may be carried out so as to bring the 40 level of solids in the whey permeate hydrolysate condensate to within a range of from about 65 to about 80 percent solids, preferably 70-75 percent solids.

Non-Settling Hydrolyzed Whey Permeate

The present invention also includes a liquid whey permeate hydrolysate condensate composition made in accordance with the method of the present invention.

The present invention includes a liquid whey permeate hydrolysate condensate composition comprising a non-settling hydrolyzed whey permeate, wherein the whey permeate hydrolysate has a degree of hydrolysis in the range of from about 65 percent to about 80 percent, contains solids within a range of from about 65 to about 80 percent solids, preferably 70-75 percent solids, and resists settling for at least 2 weeks when maintained at 90 degrees F. The liquid 55 composition also has a viscosity in the range of from about 20 milli-Pascals (or centipoise) at 90 degrees F. to about 120 milli-Pascals (or centipoise) at 5 degrees F. as measured by a Brookfield Viscometer.

Method of Producing a Dry Product from Non-Settling 60 Hydrolyzed Whey Permeate

The present invention also includes a method of producing a dried product from a non-settling hydrolyzed whey permeate with an enzyme, the method comprising the steps as described above and further adding a drying agent to the 65 whey permeate hydrolysate so as to obtain a dry product. The drying agent may be any substance appropriate to the

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desired nutritional application, and examples are those selected from the group consisting of maltodextrins and starches.

As to the physical characteristics that make the liquid compositions of the present invention beneficial in terms of being able to be pumped and poured, these compositions have a viscosity in the range of from about 90 to about 130 centipoise at 90 F.°.

Nutritional Supplements, Components, Food Products and Related Methods

The method of the present invention allows the production of a "milk syrup" liquid which is a pumpable, pourable, non-settling liquid preferably at 75-80% solids and which contains hydrolyzed lactose components and milk minerals.

The present invention also allows for the production of a dry product with same composition as the liquid concentrate produced in accordance with the present invention. This may be done with the aid of the addition of a drying aid, such as maltodextrin, starch or other well known drying aids. The present invention therefore includes methods of producing nutritional supplements, compositions and foodstuffs using the dried form of the liquid product composition of the present invention, the compositions themselves, and methods of their use.

The dry product composition of the present invention may be used in place of a corn syrup solids replacement for ice cream and other food applications. The present invention includes methods of producing nutritional supplements, compositions and foodstuffs using the liquid product composition of the present invention as a corn syrup solids substitute, the compositions themselves, and methods of their use.

The liquid product composition of the present invention may be used as a brown rice syrup replacement in foods products as is known in the art, such as for nutrition/protein bars and the like which offer a reduced glycemic index relative to sucrose or corn syrup solids. Examples of brown rice syrup uses include use as a sweetener, or for making baked goods such cookies, crisps, granola, pies, and puddings, and may be combined with another sweetener such as maple for cakes. Thus, the present invention includes methods of producing nutritional supplements, compositions and foodstuffs using the liquid product composition of the present invention as a brown rice syrup substitute, the compositions themselves, and methods of their use.

The liquid product composition of the present invention also may find beneficial application as a molasses replacement in nutritive compositions and formulations, such as for animal feed applications. Accordingly, the present invention includes methods of producing nutritional supplements, compositions and foodstuffs using the liquid product composition of the present invention, the compositions themselves, and methods of their use.

The liquid product composition of the present invention also may be used as a liquid rumen microorganism stimulant in the same manner as described for a corresponding dry product, as described in U.S. Pat. No. 6,033,689, which is hereby incorporated herein by reference. Accordingly, the present invention includes methods of producing such a liquid rumen microorganism stimulant and methods of its use for stimulating the growth of microorganisms in a ruminant animal by administering to the ruminant animal an effective amount of a liquid composition according to claim

The liquid product composition of the present invention also may be used as a pelleted feed improvement in which

the NHWP acts as a binder in place of those as applied in accordance with known formulations and processes.

The liquid product composition of the present invention also may be applied as an agglomeration aid for fast dispersing dried milk replacement products in a wide variety of forms and for several applications, such as a natural dairy beverage additive in the form of agglomerated natural milk powder as described in U.S. Pat. No. 6,777,014, incorporated herein by reference. The invention thus includes a fast dispersing dried milk replacer product comprising an agglomeration aid comprising a liquid composition according to the present invention.

The liquid product composition of the present invention also may be used in a protein and carbohydrate encapsulated fat composition comprising an encapsulant component, wherein the encapsulant component encapsulant component comprising a liquid composition according to the present invention. The invention therefore includes a protein and carbohydrate encapsulated fat protein and carbohydrate encapsulated fat made using a liquid composition according to the present invention. Such compositions may be used as calf milk replacers, and the invention also includes a method of providing such nutrition to calves.

Although not limited to the theory of the invention, it is believed these improved properties are a result of the 25 processing conditions used to enzymatically treat the whey permeate and concentrating the whey permeate condensate.

The methods of the present invention may be practiced using lactose as an alternative starting material as also described in co-pending patent application entitled NON-SETTLING GALACTOOLIGOSACCHARIDE-RICH LIQUID CONCENTRATE AND RELATED METHODS AND NUTRITIONAL COMPOSITIONS filed Mar. 22, 2010, hereby incorporated herein by reference.

BRIEF DESCRIPTION OF THE DRAWINGS

- FIG. 1 is a schematic flow diagram of a reaction process in accordance with one embodiment of the present invention;
- FIG. 2 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 3 is a graph of sugars versus reaction time showing 45 a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 4 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. **5** is a graph of lactose versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the 55 present invention.
- FIG. 6 is a graph of galactooligosaccharides (GOS) versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 7 is a photograph showing the physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. **8** is a table showing the measurements of chemical properties from HPLC data taken from whey permeate 65 hydrolysate in accordance with one embodiment of the present invention.

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- FIG. 9 is a table showing the details of a process for producing a molasses substitute material in the form of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 10 is a table showing a trial process for demonstrating the effect of hydrolysis on permeate solids in the production of whey permeate hydrolysate concentrates in accordance with one embodiment of the present invention.
- FIG. 11 is a table showing the details of a process and experimental design for producing whey permeate hydrolysate concentrates in accordance with one embodiment of the present invention.
- FIG. 12 is a table showing the data from several experiments for demonstrating the physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention, and includes a table showing a typical sugar test sample preparation from several experiments for demonstrating the chemical and physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 13 is a table showing the data from several experiments involving varying enzyme concentrations, for demonstrating the physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 14 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 15 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 16 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 17 is a table showing the data from several experiments involving varying reaction times and showing physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 18 is a table showing the data from several experiments involving varying reaction times and showing physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 19 is a table showing the data from several experiments involving varying reaction times and showing physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 20 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 21 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 22 is a graph of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
 - FIG. 23 is a graph of lactose versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.

- FIG. 24 is a graph of GOS versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 25 is a table showing a trial process for demonstrating the effect of hydrolysis on permeate solids in the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 26 is a graph of lactose hydrolysis versus time for various enzyme concentrations for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 27 is a graph of percent lactose hydrolysis versus time for various enzyme concentrations for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. **28** is a table showing the data from several experiments involving varying reaction times and showing physical properties of whey permeate hydrolysate in accordance 20 with one embodiment of the present invention.
- FIG. 29 is a graph of lactose hydrolysis versus time for various enzyme concentrations for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 30 is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. **31** is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with ³⁰ one embodiment of the present invention.
- FIG. 32 is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. **33** is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 34 is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. 35 is a graph of viscosity versus solids in the production of whey permeate hydrolysate concentrate in accordance with one embodiment of the present invention.
- FIG. **36** is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with 45 one embodiment of the present invention.
- FIG. 37 is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.
- FIG. **38** is a table showing the data from several experiments detailing the settling profile and showing physical properties of whey permeate hydrolysate concentrate in accordance with one embodiment of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In accordance with the foregoing summary of the invention, the following presents a detailed description of the preferred embodiments, which are considered to be the best 60 mode thereof.

The preferred method and compositions described herein are not intended to be exhaustive or to limit the invention to the precise forms disclosed. They are chosen and described to explain the principles of the invention and the application 65 of the method to practical uses so that others skilled in the art may practice the invention.

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Example 1 of the Manufacturing Process of the Present Invention

As a preferred but non-limiting example of the method by which compositions of the present invention may be made, the following steps may be followed:

FIG. 1 is a schematic flow diagram of a reaction process, and is representative of the process to manufacture a non-settling hydrolyzed whey permeate (NHWP) in accordance with one embodiment of the present invention.

The process steps for manufacture of NHWP include:

- 1. Add whey permeate or lactose to hydrolysis vessel (15-25% solids, preferably 18-20%)
- 2. Heat whey permeate or lactose solution to 70 to 80° C. (preferably 75° C.) for 15 to 45 min (preferably 30 min)
- 3. Cool to specified reaction temperature of enzyme used (different for different enzyme sources)
- Hydrate enzyme in process water at reaction temperature in separate vessel and transfer to hydrolysis vessel to start hydrolysis
- 5. Carry out hydrolysis at optimal temperature (25 to 50° C.—preferably 35-40° C.) and pH optimal (5.0 to 7.5—preferably 6.5-7.0) for 3 to 24 hours (preferably 12 to 16 hours)
- After hydrolysis has reached desired level transfer to evaporator and evaporate to 60 to 80% solids (preferably 75-80% solids)
- 7. When desired level of solids is reached transfer to product storage vessel

Detailed lab and plant protocols along with HPLC sugar profiles are attached as the Figures hereto.

As can be appreciated from FIG. 1, this schematic shows a flow diagram of a reaction process in accordance with one embodiment of the present invention. The whey permeate is preferably held in a holding tank with available stirring as shown. Likewise, the enzyme, such as Novo® Lactase (commercially available from Novozymes of Bagsvaerd, Denmark) or Validase® (commercially available from Valley Research of South Bend, Ind.), is held in a holding tank with available stirring. The whey permeate and enzyme are conducted to a hydrolysis reaction tank where hydrolysis takes place under the above described conditions. The resultant hydrolysate is then conducted to an evaporator where it is concentrated to the solids level described herein. The resultant hydrolysate concentrate may then be further conducted to a product storage tank or through conduits for further processing or packaging as required by the desired application.

FIGS. **2-4** are graphs of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention, showing the results for various respective Novo Lactase concentrations.

FIG. **5** is a graph of lactose versus reaction time showing a typical reaction profile for the production of whey permeste hydrolysate in accordance with one embodiment of the present invention, showing the results for various respective Novo Lactase concentrations.

FIG. 6 is a graph of galactooligosaccharides (GOS) versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention, showing the results for various respective Novo Lactase concentrations.

FIG. 7 is a photograph showing the physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention, having 80% solids and after 12 hours hydrolysis at pH 6.5 with various respective Novo Lactase concentrations.

FIG. **8** is a table showing the measurements of chemical properties from HPLC data taken from whey permeate hydrolysate in the processes described herein.

FIG. **9** is a table showing the details of a process for producing a molasses substitute material in the form of whey permeate hydrolysate concentrate as described herein.

FIG. 10 is a table showing a trial process for demonstrating the effect of hydrolysis on permeate solids in the production of whey permeate hydrolysate concentrates in accordance with one embodiment of the present invention.

FIG. 11 is a table showing the details of a process and experimental design for producing whey permeate hydrolysate in accordance with one embodiment of the present invention.

FIG. 12 is a table showing the data from several experiments for demonstrating the physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention, and includes a table showing a typical sugar test sample preparation from several experiments for demonstrating the chemical and physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.

FIG. 13 is a table showing the data from several experiments involving varying enzyme concentrations, for demonstrating the physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.

FIGS. 14, 15 and 16 are graphs of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with several embodiments of the present invention. These graphs elucidate the properties that give rise to the preferred embodiment of the invention.

FIGS. 17, 18 and 19 are tables showing the data from several experiments involving varying reaction times and showing physical properties of whey permeate hydrolysate in accordance with several embodiments of the present invention, and serves to show the variations in the preparation of whey permeate hydrolysate in accordance with the invention.

FIGS. 20, 21 and 22 are graphs of sugars versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate with differing enzyme concentrations, in accordance with several embodiments of the present invention.

FIG. 23 is a graph of lactose versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance with several embodiments of 50 the present invention.

FIG. 24 is a graph of GOS versus reaction time showing a typical reaction profile for the production of whey permeate hydrolysate in accordance several embodiments of the present invention.

FIG. 25 is a table showing a trial process for demonstrating the effect of hydrolysis on permeate solids in the production of whey permeate hydrolysate in accordance with several embodiments of the present invention.

FIGS. **26** and **27** are graphs of lactose hydrolysis versus 60 time for various enzyme concentrations for the production of whey permeate hydrolysate in accordance with several embodiments of the present invention.

FIG. **28** is a table showing the data from several experiments involving varying reaction times and showing physical properties of whey permeate hydrolysate in accordance with one embodiment of the present invention.

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FIG. 29 is a graph of lactose hydrolysis versus time for various enzyme concentrations for the production of whey permeate hydrolysate in accordance with one embodiment of the present invention.

FIGS. **30-34** are graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with several embodiments of the present invention.

FIG. **35** is a graph of viscosity versus solids in the production of whey permeate hydrolysate concentrate in accordance with several embodiments of the present invention. This graph shows the viscosity levels that may be achieved in accordance with the present invention.

FIG. **36** is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with several embodiments of the present invention.

FIG. 37 is a graph of enzymatic hydrolysis in the production of whey permeate hydrolysate in accordance with several embodiments of the present invention.

FIG. 38 is a table showing the data from several experiments detailing the settling profile and showing physical properties of whey permeate hydrolysate concentrate in accordance with several embodiments of the present invention. This graph shows the beneficial settling profiles that may be achieved in accordance with the present invention.

The characteristics of the NHWP prepared in accordance with this embodiment of the present invention include the ability to concentrate a hydrolyzed whey permeate to a pumpable, pourable, non-settling liquid, most preferably with 70-80% solids.

The method of the present invention allows the production of a "milk syrup" liquid which is a pumpable, pourable, non-settling liquid preferably at 75-80% solids and which contains hydrolyzed lactose components and milk minerals.

The present invention also allows for the production of a dry product with same composition as the liquid concentrate produced in accordance with the present invention. This may be done with the aid of the addition of a drying aid, such as maltodextrin, starch or other well known drying aids. The present invention therefore includes methods of producing nutritional supplements, compositions and foodstuffs using the dried form of the liquid product composition of the present invention, the compositions themselves, and methods of their use.

The dry product composition of the present invention may be used in place of a corn syrup solids replacement for ice cream and other food applications. The present invention includes methods of producing nutritional supplements, compositions and foodstuffs using the liquid product composition of the present invention as a corn syrup solids substitute, the compositions themselves, and methods of their use.

The liquid product composition of the present invention may be used as a brown rice syrup replacement in foods products as is known in the art, such as for nutrition/protein bars and the like which offer a reduced glycemic index relative to sucrose or corn syrup solids. Examples of brown rice syrup uses include use as a sweetener, or for making baked goods such cookies, crisps, granola, pies, and puddings, and may be combined with another sweetener such as maple for cakes. Thus, the present invention includes methods of producing nutritional supplements, compositions and foodstuffs using the liquid product composition of the present invention as a brown rice syrup substitute, the compositions themselves, and methods of their use.

The liquid product composition of the present invention also may find beneficial application as a molasses replacement in nutritive compositions and formulations, such as for

animal feed applications. Accordingly, the present invention includes methods of producing nutritional supplements, compositions and foodstuffs using the liquid product composition of the present invention, the compositions themselves, and methods of their use.

The liquid product composition of the present invention also may be used as a liquid rumen microorganism stimulant in the same manner as described for a corresponding dry product, as described in U.S. Pat. No. 6,033,689, which is hereby incorporated herein by reference. Accordingly, the present invention includes methods of producing such a liquid rumen microorganism stimulant and methods of its use.

The liquid product composition of the present invention also may be used as a pelleted feed improvement in which 15 the NHWP acts as a binder.

The liquid product composition of the present invention also may be applied as an agglomeration aid for fast dispersing dried milk replacement products in a wide variety of forms and for several applications.

While specific formulations and process steps are discussed, it should be understood that this is done for illustrative purposes only. A person skilled in the relevant art will recognize that other process and composition variations can be used without departing from the spirit and scope of the 25 invention. It will be apparent to a person skilled in the relevant art that this invention can also be employed in a variety of other applications.

What is claimed is:

1. A method of producing a non-settling hydrolyzed whey permeate with an enzyme, the method comprising the steps: subjecting the whey permeate having an initial solids content in the range of from about 15 to about 25 12

percent solids to hydrolysis by an enzyme, so as to obtain a whey permeate hydrolysate having a degree of hydrolysis above about 65 percent; and

subjecting the whey permeate hydrolysate to evaporation so as to bring the level of solids in the resulting whey permeate hydrolysate concentrate to within a range of from about 60 to about 80 percent solids, so as to obtain a whey permeate hydrolysate concentrate whose settling profile is such that there is no detectable settling over two weeks when stored at 90° F.

- 2. A method according to claim 1 wherein the whey permeate has an initial solids content in the range of from about 18 to about 20 percent solids.
- 3. A method according to claim 1 wherein the hydrolysis by the enzyme is carried out for sufficient time to bring about a degree of hydrolysis between about 65 percent and about 80 percent.
- 4. A method according to claim 1 wherein the evaporation is carried out so as to bring the level of solids in the whey permeate hydrolysate to within a range of from about 70 to about 75 percent solids.
 - 5. A method according to claim 1 wherein the resulting whey permeate hydrolysate concentrate has a viscosity in the range of 90 to 120 centipoise at 90° F.
 - **6.** A method according to claim **1** wherein the resulting whey permeate hydrolysate concentrate has a galactooligosaccharides (GOS) content in the range of about 3% to 5% by weight.
 - 7. A method according to claim 1 further comprising adding a drying agent to the whey permeate hydrolysate.
 - **8**. The method of claim **7** wherein the drying agent comprises a maltodextrin or a starch.

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